

Non-traditional grains in low and high moisture extrusion applications –Residence time,
physico-chemical properties and resistant starch

by

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Abstract

Sorghum, millets and teff are important staple crops worldwide, especially in semi-arid regions of Africa and India because of their drought tolerance. However, lack of research and other limitations have restricted their usage in food products. This study is focused on extrusion processing of low and high tannin sorghum varieties, millet and teff for high and low moisture applications, and to investigate process characteristics such as residence time distribution and specific mechanical energy, physico-chemical properties of resultant food products (such as pre-cooked pasta and expanded snacks) and their resistance starch content. Results from preliminary lab scale extrusion, including optimization of starch type and level for pre-cooked pasta and in-barrel moisture for expanded snacks, were used to design pilot scale studies on a twin-screw extruder.

In the first pilot-scale experiment, decorticated white sorghum blends prepared with addition of mono-glycerides (0.5%, 1% and 1.5%) and salt (1%) were processed at three different in-barrel moisture contents 40%, 44% and 48% (wet basis) for processing of precooked pasta. The optimum formulation containing 1% mono-glycerides and process conditions corresponding to 48% in-barrel moisture were also used to develop precooked teff and millet pasta. The non-traditional grain based pastas were investigated for cooking quality, thermal characteristics using differential scanning calorimeter, pasting properties using rapid visco analyzer and texture profile analysis. In general, increasing in-barrel moisture led to reduction in solid losses (ranging from 4.0-8.2% for all treatments), indicating improvement in cooking quality. On the other hand, increase in mono-glycerides concentration led to higher cooking losses, and also affected pasting and textural properties significantly. Sorghum-based precooked pasta was of best quality while millet pasta was poorest in cooking quality, and visual and textural attributes. Cooking loss for control pre-cooked pasta produced in this experiment using semolina was 4.5%, and commercial semolina pasta was 3.2%.

Residence time distribution in pilot-scale twin screw extruder, during high moisture process conditions used for pre-cooked pasta, was also investigated at three different in-barrel moistures (40%, 44% and 48%) and monoglycerides/ lipid (0.5%, 1% and 1.5%) concentrations. Increase in in-barrel moisture significantly decreased mean residence time. For example, mean residence time was 4.47 mins at 40% moisture, 3.89 mins at 44% and 3.74 mins at 48%. On the contrary, residence time significantly increased with lipid level. For example, mean residence time was 3.87 mins at 0.5% concentration of mono-glycerides, 4.48 mins at 1% and 4.70 mins 1.5%.

In the second experiment focusing on low moisture applications, pilot-scale twin screw extrusion was used to process decorticated white sorghum and high tannin sumac sorghum for expanded snacks. The addition of sumac bran decreased the specific mechanical energy input (366-578 kJ/kg) and expansion ratio (6.4-7.9), and resulted in higher piece density of extrudates. Use of sumac bran and sumac flour led to increase in resistant starch content, although it was less than 1% for all treatments. Therefore, extrusion with ingredients having high tannin content does not provide value, despite tannins being associated with resistant starch at least in raw materials.

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Chapter 1 - Introduction

The global gluten-free (GF) packaged food market is projected to grow at a compound annual growth rate of approximately 6% between 2015 and 2019. As per a report published by Transparency Market Research gluten free food market is projected to grow at 7.7% from 2015 to 2021. In terms of volume, the global gluten free food market was valued at 277.2 ('000) metric tons in 2014 and is expected to reach 423.0 ('000) metric tons by 2021, expanding at a CAGR of 6.1% over the forecast period.

As the market for gluten-free processed foods grows every day and has proven to be new evolution in food industries despite its challenges. New products enter stores every day whether it's a new application or simply another company seeking to ride the trend's rising popularity. Gluten-free consumers are now presented with cakes, pizzas, noodles, pasta, snacks, breakfast cereals and sweet goods suitable for their intolerances or chosen diets — a far cry from when bread was the only gluten-free product on the shelf. The consumer is getting more choices and people with celiac disease will be having more products to eat. Gluten-free products have evolved, and with that evolution, nutrition, texture and taste are more important than ever. Their use has been promoted and recommended in various dietary guidelines either as whole grain or processed food. Gluten-free products have turned from being medicated products for gluten intolerant people to a lifestyle choice across all customer segments. This growth engine has brought attention to alternate grains such as sorghum, teff, quinoa and millets etc., to play a major role in food processing.

There are a variety of GF whole grains available, each with its own unique texture and flavor including corn, millet, oats, brown rice, sorghum, teff, millet, amaranth and quinoa. Some of the gluten free grains are superior in nutritional value over traditional grains such as wheat, corn or rice. Teff, millet and sorghum are among such grains that remained unexplored especially teff and millet. In this research white decorticated whole sorghum, ivory (white) teff and proso millet grain were studied.

Gluten Free Foods and Health Benefits

GF food products offer alternative food choices to people that are gluten intolerant. Celiac disease is a lifelong intolerance to two protein fractions, gliadin of wheat and the prolamins of rye (secalin), barley (hordeins) and possibly oats (avidins) (Murray, 1999). The ingestion of gluten can cause inflammation of the small intestine leading to the malabsorption of several important nutrients including iron, folic acid, calcium and fat-soluble vitamins, and small intestine functionality becomes severely impaired (Feighery, 1999; and Murray, 1999). Murray (1999) concluded that celiac disease is the result of three processes (genetic predisposition, environmental factors and immunologically based inflammation) that culminates in intestinal mucosal damage. The only effective treatment for coeliac disease is a strict adherence to a gluten-free diet throughout the patient's lifetime, which, in time results in clinical and mucosal recovery.

Gluten is the main structure-forming protein in flour, and is responsible for the elastic characteristics of dough, and contributes to the appearance and crumb structure of many products. Gluten removal results in major problems for bakers, and currently, many gluten-free products available on the market are of low quality, exhibiting poor mouthfeel and flavor (Arendt et al., 2002). This presents a major challenge to the cereal technologist and baker alike, and has led to the search for alternatives to gluten in the manufacture of gluten-free bakery products. However, gluten is not a structure forming component in extruded products. Extrusion is high thermal and high shear process with a moderate range of moisture (10% to 40%). High shear breaks down starch at molecular level which forms structure once fluid melt releases through die. To define and optimize process to improve physico-chemical characteristics of gluten free (GF) pasta still represents a challenge for researchers and industry. For precooked pasta making several ingredients (modified starch, GF flours, and additives) have been used as alternative to gluten to create a starch based network than can withstand the physical stress of cooking and provide firmness to the cooked product. Moreover, various technologies have been proposed such as repeated heating and cooling steps, which are difficult to control and costly. This research work is based to study how precooked pasta can be made based three GF flours with different kinds of starches at

different levels with mono-glycerides and how they impact physico-chemical properties of precooked pasta.

Pasta

Pasta is considered as one of the simplest cereal based products in terms of based ingredients (semolina, and water) and processing (includes hydration, mixing, forming and frying steps). Both raw material and processing conditions play a critical role in determining the quality of final pasta. Pasta basically originated in Italy but is a most famous food in the world. The cooking convenience, palatability, long and stable shelf life and nutritional properties are key eye catchers. The term usually refers to unleavened extruded wheat dough, composed simply of flour and water, some- times egg. A significant part of the human population, however, cannot tolerate gluten, a protein composite found in wheat, rye and barley. Hence, it is necessary to develop products based on alternative cereals or pseudo-cereals. Also for the non-coeliac population, switching from refined wheat products to nutritionally more valuable grains could bring benefits regarding health and well-being. In last few decades, a new group of categories, the gluten free (GF) pasta has grown rapidly not only by the growing number of celiac but also by regular consumers who prefer GF pasta for nutritional benefits. One of the objectives of this study was to develop optimum quality sorghum, teff and millet pasta comparable to traditional wheat pasta.

Pasta Manufacturing

Gluten protein and starch are two key components important in pasta manufacture both in precooked pasta and raw pasta. The gluten protein bodies in durum wheat are present in wedge shaped structures between the voids starch granules. Gluten protein is a glassy material in dry state, but the addition of water makes it rubbery and elastic. It acquires the ability to form strands and sheets through intermolecular bonds. These properties make gluten essential to its role as the continuous matrix which traps and encapsulates starch in pasta and holds product shape during manufacture and cooking. Heating of protein bodies leads to irreversible formation of protein-protein crosslinks. It

is important to control protein heating in pasta manufacturing to stabilize structure and texture of pasta.

Major component of semolina is starch (85%). Starch act as filler, raw starch has limited water absorption capacity below 55°C. Upon heating, starch loses its crystalline structure and can absorb high amount of water. The water absorption cause swelling of granules followed by viscosity development till it disintegrates and dissolves into suspension. The structural expansion of pasta raw materials can be understood by the concept of glass-rubber transition. Kalichevsky et al. (1992) and Kokini et al. (1994) explained that at 10% moisture content and 25°C prior to wetting and extrusion, gluten protein exists in glassy state. A more usual moisture content of 13%-15% at the same temperature will result in the gluten which has undergone the glass transition and has some of the rheological properties of a rubbery material. As water percent is raised to 33% during wetting, it will start developing the properties of a flexible material with the ability to flow under applied stress. This is the desired state to make high-quality pasta with optimum texture and mouthfeel.

The first stage in pasta forming includes the dampening of dry raw material with liquid ingredients. To produce good quality pasta, it is very important to achieve optimum semolina hydration. The hydrated semolina is fed to extruder to form dough and give a desired shape. Extrusion processing is combines factors such as mixing, kneading, pressure and temperature and cooking results into developing physical structure of pasta. The extrusion application involves repeated heating and cooling treatments induces starch gelatinization and retrogradation phenomena, creating starch network capable of standing up to cooking stresses (Pagani, 1986). The comprehensive character of the extrusion cooking technique provides the option of modifying the extruder by changing the configuration of the screws, the use of cooling or heating segments of the extruder and the application of various shape forming dies. Due to thermal and pressure treatment, instant pasta is already precooked and requires only rehydration in boiling water or short cooking (Kruger et al.,1996).

Precooked pasta is usually made by twin screw extrusion cooking technique. The

process requires addition of water and steam during pasta processing. The dough inside extruder barrel passes through low shear and gentle screw mixing at different temperatures. The temperature ranges are between 50°C to 70°C (Wang et al., 1999). An extremely important parameter in the extrusion process is the dough moisture content. When manufacturing simple pasta forms such as threads or spaghetti type, the dough moisture may be relatively low around 28%-29%, while for products with more complicated shapes, it is necessary to ensure higher dough moistening up to 32% (Wojtowicz, 2006). Optimum moisture allows smooth flow of dough inside barrel whereas excessive dough moisture decrease degree of gelatinization results into unstable structure and high cooking losses post drying. Low dough moisture lead to expansion and results into developing undesirable bristles. The extrusion system was usually equipped with vacuum system to take out steam form barrel and cool the cooked melt to form a condensed pasta structure. It is very important to cool the cooked melt to avoid bubble formation in pasta matrix. Pasta with bubbles in structure disintegrates quickly during cooking results into high cooking loss (Wojtowicz et al., 2009). Once the product is out from extruder die it is dried at low temperature for longer time to reach the moisture level of 12.5%. Usually the drying of pasta was carried out at 70°C to 75°C for 60 mins, high temperatures are avoided to prevent cracking of pasta structure. Starch in a processed product is approximately 90% gelatinized, the level of microbiological contamination is low and the ability for rapid rehydration is high, even in cold water (Manthey et al., 2004).

The role of raw materials is also very important. The technology developed by the Wenger Company (USA) shows the characteristic composition of extrusion cooked pasta of firm consistency is: 98% semolina, 1% mono-glyceride, 1% powdered egg albumin. The soft texture of pasta can be obtained using semolina (98.5%) and mono-glycerides (1.5%). Protein, gluten, eggs, milk or emulsifiers are added to reduce the hydration time. On the other hand, the addition of mono-glycerides reduces the adhesiveness of pasta made from soft wheat flour and can also influence the quantity of cooking losses (Malcolmson and Matsuo, 1993). This study is aimed to quantify both process moisture conditions and mono-glycerides to develop high-quality pasta by using sorghum, teff and millets.

Residence Time Distribution

The second objective of this study was to determine the residence time distribution (RTD) at three different in-barrel moistures (40%, 44% and 48%) and mono-glycerides concentrations (0.5%, 1% and 1.5%) used in extrusion process. RTD is a measure of the length of time feed materials spends in continuous extruder flow system. Apart, from feed material properties, in-barrel moisture and lipid levels also affects the mean residence time, the residence time distribution (RTD), and the flow patterns in extruder. The effect of moisture content on the mean residence time for cereal processing in twin extrusion was studied by (Liang et al., 2012; Kumar et al., 2008 and Sisay et al., 2017). Increase in in-barrel moisture (19%) reduced both mean residence time and RTD spread with reduced gluten based formulation in twin screw extruder (Sisay et al., 2017). Chen et al. (2010) found shorter residence time at high in-barrel moisture content for soy protein processed in twin screw extruder. However, various studies have shown opposite results at similar moisture levels. Kumar et al. (2008) reported increase in mean residence time with increase in in-barrel moisture content from 16 to 28% for native starch processed in twin screw extruder. Altomare and Ghossi (1986) found increase in moisture content from 10% to 28.4% increased residence time from 21 to 25 seconds in twin screw extruder.

Similarly, the inclusion of lipids into the formulation or into the barrel also affects the RTD. The addition of lipids develops slippage, which not only reduces the energy input but also increase the mean residence time and RTD spread. Increase in lipid levels significantly ($p < 0.05$) increased the mean residence time of rice flour for increased fish solid concentration (5%, 10% and 15%) (Choudhury and Gautam, 2003). Similar results of higher residence time were reported by Phillips and Facone (1988), for sorghum meal processing at 15% and 30% peanut fat addition. This study was dedicated to confirming the effect of in-barrel moisture and lipid concentrations on residence time in a low shear pasta making process.

Sorghum Tannin Based Expanded Snack and Resistant Starch

The growing market of low calorie processed snack food worldwide had surged the food processing industries interest to dig deep into the functional properties of various cereal. Sorghum is a cereal crop grown in semi-arid regions around the world. Its resistance to drought and heat makes it an ideal crop for regions faced with the threat of climate change and global warming (IPCC, 2007; Srivastava et al., 2010). There is growing interest in the food industries worldwide for the use of sorghum for its potential health benefits related to slow starch digestibility, high antioxidant and phenolic properties. Sorghum has similar starch content to wheat and maize, but the presence of polyphenolics, unique protein matrix and tannins have made it a very special grain. The presence of polyphenols and tannins has been shown to lower starch digestibility and overall calorie intake in sorghum foods (Taylor and Emmambux, 2010). Jenkins et al. (1981) and Buyken et al. (2010), reported that foods high in slow digestible starch (SDS) reduce the risk of chronic disease, especially of type 2 diabetes mellitus. Sorghum based diets containing phytochemicals delivered several health benefits such as potential to reduce risk of cardiovascular disease and certain types of cancer in humans (Awika and Rooney, 2004).

The supplementation of wholegrain sorghum flour have reduced starch digestibility and increased the antioxidant capacity of refined wheat flour flat bread (Yousif et al., 2012). The addition of wholegrain sorghum flour increased resistant starch content and antioxidant activity of durum semolina pasta (Khan et al., 2013). Henceforth, in societies where over-nutrition leading to obesity and type- 2 diabetes mellitus is a chronic concern, sorghum grain has the potential to be used to maintain slow starch digestion and high antioxidant capacity in food products and provide protection against such chronic diseases.

The interactions of sorghum tannins (proanthocyanidins) with starch molecules have increased resistant starch in high amylose starch formulation by 52% higher over the control. Sorghum tannins interact strongly with starch that decreases the starch digestibility (Barros et al., 2012). Higher molecular weight phenolic extracts when

formulated with higher amylose starch increased the levels of resistant starch content. Sorghum tannins have the natural potential to modify starch by interacting strongly with amylose and are thus most suitable to produce foods with higher resistant starch (Barros et al., 2013). Therefore, the third objective of this study was to quantify resistant starch formation in sorghum tannin based expanded snack processed in twin screw extrusion.

Scope of This Study

Focus: Process conditions and formulations optimized on lab scale extrusion can be used to effectively scale up to pilot scale.

Objective 1: Quantification of process moisture and mono-glycerides levels to develop a high-quality sorghum, teff and millet pasta.

Focus: Optimal energy input based on in-barrel process moisture and lipid (mono-glycerides) concentration can lead to the desired starch transformation to form a strong protein- starch matrix in the absence of gluten, leading to good quality gluten-free product. Plasticizing and lubricating effects of water and mono-glycerides respectively can significantly impact residence time distribution in high moisture extrusion applications.

Objective 2: To study the extruder barrel residence time distribution (RTD) at three different in-barrel moistures (40%, 44% and 48%) and mono-glycerides (0.5%, 1% and 1.5%) concentrations for precooked sorghum pasta.

Focus: High tannin content in sorghum bran can result in increased resistant starch content in extruded products. Hence, the study was aimed to validate resistant starch retention in expanded extruded snacks formulated with sumac flour and sumac bran containing high concentrations of tannins.

Objective 3: To measure resistant starch retention in sorghum expanded snack formulated with sumac flour and condensed sumac tannins processed at different processing conditions in twin screw extruder.

Proximate analysis of raw materials

The proximate composition of grain flours used in this study was analyzed in SDK laboratories. The compositions of the individual flour used in the study are shown in Table 1.1. It can be inferred from the table that corn flour has the highest total starch content (78.2%) followed by decorticated white sorghum flour (72.9%), millet flour (70.2%), whole sumac flour (68.0%), durum wheat semolina (65.7%), teff flour (64.8%), and sumac bran (34.0%). The starch content was lower in blends with whole flour as component of whole flours had lower starch content compared to decorticate or degermed flours. The condensed sumac bran had the highest crude fiber content (5.3%), followed by teff flour (1.8%), whole sumac flour (0.9%), millet flour (0.8%) and decorticated white sorghum flour (0.5%). The fat content was highest in condensed sumac bran (6%), followed by millet flour (4.1%), whole sumac flour (2.7%), teff flour (2.0%), and decorticated white sorghum flour (1.8%). The durum wheat semolina had the highest protein content (13.8%), followed by teff flour (11.7%), millet flour (10.4), decorticated white sorghum flour (9.7%) and least for sumac flour (0.2%).

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Table 1.1: Proximate analysis of different raw materials.

Samples	Crude protein (%)	Crude fiber (%)	fat (%)	Ash (%)	Total starch (%)
Semolina	13.8±0.0	<0.2±0.0	1.1±0.0	0.7±0.0	65.7±0.4
Sorghum flour	09.7±0.0	0.5±0.1	1.8±0.1	0.8±0.0	72.9±0.0
Sumac flour	00.2±0.0	0.9±0.1	2.7±0.3	1.0±0.1	68.0±0.4
Sumac bran	00.8±0.0	5.3±0.0	6.0±0.1	3.6±0.1	34.0±0.4
Teff flour	11.7±0.1	1.8±0.2	2.0±0.0	1.9±0.0	64.8±0.4
Millet flour	10.4±0.5	0.8±0.2	4.1±0.1	1.2±0.0	70.2±0.5
Corn flour	05.0±0.0	<0.2±0.0	1.9±0.0	0.5±0.0	78.2±0.2

Chapter 2 - Millet and teff based gluten free pasta formulated with corn starch and mono glycerides as texture enhancers

Abstract

Food allergies and food intolerances are a growing public health concern causing higher consumer demand of new food products that are custom-made to meet special dietary requirements. Especially, the rapidly growing gluten free (GF) cereal product market. In this study, Ivory teff (*Eragrostis tef*) and proso millet (*Panicum miliacuem*) were utilized as base material to produce GF precooked pasta. Extrusion technology was used to process precooked pasta formulations. Native and high amylose cornstarch and mono-glycerides (MG) at different levels were used to enhance structural and textural attributes. Rice based commercial pasta as gluten free control, lab cooked durum wheat semolina and commercial rotini pasta was used as overall control. The resulting products were characterized for texture profile analysis (TPA), cooking loss, water absorption (WA), and gelatinization and viscoelastic properties. Wheat precooked pasta was of superior characteristics such as firmness, water absorption and low solids loss followed by rice, teff and millet.

The addition of MG with corn starch reduce the cooking loss of both teff and millet pasta. The native corn starch addition worked better over high amylose corn starch in controlling cooking losses. The inclusion of corn starch increased starch content of the formulations, higher starch content led to strong cooked starch matrix of pre-cooked pasta. Whereas the addition of high amylose corn starch led to leaching of amylose resulted in higher cooking loss. The twenty percent addition of native corn starch significantly ($p < 0.05$) reduced solids loss by 23.01% for teff and 37.18% for precooked millets. Addition of corn starch increased the water absorption during cooking, teff absorbed more water than millet at same addition level of corn starch. The increase in MG percentage from 0.5% to 1% resulted in higher cooking losses due to less cooking of starch. Gelatinization temperature for teff flour was in the range of 79.8°C to 81.4°C, and for millet flour it was 77.6°C to 80.5°C. Teff pasta had higher firmness values than

millet. Pasting temperature of teff ranged from 67°C to 80.3°C and for millets 59.5°C to 68.6°C. In general, increase in MG increased the peak viscosity, peak time and final viscosity of precooked pasta. The change in viscoelastic properties is the result of high shear in extrusion process. Overall lab control durum wheat pasta was of optimum quality characteristic followed by teff and millet. Teff pasta quality attributes were analogous to gluten free rice pasta group.

Key Words

Teff, Millet, corn starch, mono-glycerides, extrusion

Introduction

Transparency Market Research has published a market report titled "Gluten Free Food Market - Global Industry Analysis, Size, Share, Growth, Trends, and Forecast, 2015 - 2021. Per the report, the global gluten free food market was valued at US\$2.84 billion in 2014 and is projected to reach US\$4.89 billion by 2021, growing at a rate of 7.7% from 2015 to 2021. As the market for gluten-free processed foods grows every day and has proven to be a bandwagon that many industries are willing to jump on despite its challenges in formulating and processing. With the growing number of gluten-free products on the market, there are now more choices, and people with celiac disease will be selective in choosing what products they eat.

Pasta, once a famous food from Italy, has gaining acceptance in the world. The cooking convenience, palatability, long and stable shelf life and nutritional properties are the main characteristics. As an alternative to conventional durum wheat based pasta amaranth, buckwheat, teff, millet and quinoa precooked pasta are enriched with fiber, mineral, antioxidants and polyphenols. In last few decades, a third group of categories, the gluten free (GF) pasta has grown rapidly not only by the growing number of celiac but also by regular consumers who prefer GF pasta for nutritional benefits. Pasta is one of the 100 percentage wheat based food, where gluten protein dominates. Hence, if gluten free products need to develop it should replace pasta first as a real alternative to

gluten. Currently, there is a broad range of gluten free products based on rice, corn and other GF flours. Unfortunately, most of the GF pasta exhibits poor cooking and inferior when compared to wheat. There is a variety of gluten-free whole grains available, each with its own unique texture and flavor including corn, millet, oats, brown rice, sorghum, teff, millet, amaranth and quinoa. Some of the gluten free grains are superior in nutritional value over traditional grains such as wheat, corn or rice. Teff and millet are among such grains that remained unexplored. In this research work ivory (white) teff and proso millet grain were used. They especially are rich in minerals, iron, dietary fibers, antioxidants, phytochemicals and polyphenols, which contribute broad-spectrum positive impacts to human health.

Ivory teff (*Eragrostis tef*) is an ancient grain that has been cultivated and used for human consumption in Ethiopia for centuries. Teff is perhaps the smallest cereal grain on earth with an average length of 1mm (Umeta and Parker, 1996; Lacey and Llewellyn, 2005; Adebowale et al., 2011). The color of teff varieties varies from bright white (ivory) to dark brown (black), and nutritional quality varies accordingly.

Teff is carbohydrate rich grain (80%) with starch content of approximately 73%. The amylose content of 13 teff varieties tested ranged from 20 to 26% (Bultosa, 2007). Teff starch granules sized from 2-6 μm using scanning electron microscopy (Bultosa et al., 2002) (Figure 2.1), that makes it lower than wheat (A type 20-35 μm), sorghum (20 μm) and maize (20 μm) (Delcour et al., 2010). Given the smaller size and large surface area, smaller starch granules are more susceptible to enzymatic attack (Tester et al., 2004). The predicted glycemic index of teff (74) was found significantly lower than that of white wheat (100) but comparable to that of sorghum (72) and oats (71) (Wolter et al., 2013). The average crude protein of teff is in the range of 8 to 11 percent, the higher levels of glutamine, alanine, leucine, and proline and the comparatively lower content of lysine suggest that major prolamins are the major storage proteins (Adebowale et al., 2011). Likewise, compared to other cereals, higher contents of isoleucine, leucine, valine, tyrosine, threonine, methionine, phenylalanine, arginine, alanine, and histidine are found in teff (Bultosa and Taylor, 2004; FAO, 1992). Red teff has a higher iron and calcium content than mixed or white teff (Abebe et al., 2007). Teff is a comparatively good

source of essential fatty acids, fiber, minerals (especially calcium and iron), and phytochemicals, such as polyphenols and phytates. Teff as a rich iron source can be used as good alternative (Adish et al., 1999). Alaunyte et al., (2012) found that iron content of wheat reached more than double with 30% teff flour supplementation. Bokhari et al. (2012) showed that consumption of 30 percent teff-enriched wheat breads can help maintain serum iron levels in pregnant women. Given the high iron content of teff and its potential contribution to food-based approaches to improve nutrition is of great advantage. Teff is naturally gluten free cereal with more nutrient dense profile such as good amount of minerals, fiber and phytochemicals. The low glycemic index of teff makes it very important for diabetic patients especially with celiac disease.

Millet is the sixth largest in small grain production tonnage worldwide and thrive in sub-arid and arid regions (Paust, 2006). On a nutritional basis, millets are a balanced source of carbohydrates, proteins, fat, and major vitamins/minerals. However, their usage for human consumption is limited and primarily is utilized for poultry feed. Structurally, millet is a small grain, composed of an outer fibrous pericarp, a starchy endosperm and a germ body. Millet inherently has numerous kernel shapes, sizes and colors. Traditionally, millet is hand pounded and winnowed to separate the pericarp, germ and endosperm. The pericarp and germ removal is completed to reduce bitterness in final food forms. The subsequent starchy endosperm is then consumed in the form of porridges, flatbreads, and cereal dishes.

The corneous endosperm contains polygonal starch granules implanted in protein bodies. The amylose content ranges from 17.21% to 32.6% (Yanez et al., 1991). The protein levels of millet is in range from 11.5% to 13% with maximum of about 17%, the range vary according to the variety, environment conditions, and nutrient content of soil and water (Geervani and Eggum, 1989; Dendy, 1995; Parameswaran and Thayumanavan, 1995). Light color varieties are higher in protein level than dark colored. Dry conditions in growing season enhance the protein content but affect the protein quality adversely. (Kalinova et al., 2006). The protein content of proso millet is higher than other varieties of millet (Geervani and Eggum, 1989). Millets are also a rich source fat and fiber. The fiber content of de-hulled proso millet is in the range of (0.8%

to 1.2%) to the levels of oat (Geervani and Eggum, 1989). The soluble fiber is comprised of 36% of total fiber content. Lipids constitute a very fraction in cereals but lipid levels of de-hulled proso millet ranges from 3.5% to 6.7%. (Jones et al., 1970; Ravindran, 1991; Kalinova, 2002). The germ contains 25% of total lipids. The free, bound and structural lipid content is around 62.2%, 27.8% and 10% (Sridhar and Lakshminarayana, 1994). The proso millet lipids comprised of 86% to 89% unsaturated acids, among that 42% is poly unsaturated fatty acids (Becker, 1994). The main fatty acids of proso millet are linolenic (38.4% to 66.86%), oleic (21.4% to 22.7%) and palmitic acid (6.61% to 11.3%) (Dendy, 1995). These fatty acids very easily and give unpleasant taste to de-hulled grains when storage time is too long. Proso millet is also a rich source of vitamin B1, B2, B3, B6 and E (Dendy, 1995). The phenolic component of proso millets is about 0.05 mg and 0.10 mg per 100 gm on dry basis.

Replacement of the gluten network to produce GF products is a major technological challenge, gluten being the essential structure-building protein. Thus, substances that imitate the viscoelastic properties of gluten are always required in GF products (Mariotti et al., 2009). The first step in this direction is the utilization of starch retro-gradation. The formation of a “scaffold” is developed as alternatives to gluten networking especially when the base flours are from gluten free cereals. In order to have a good amount of retrograded starch in the pasta, it is necessary to induce starch disorganization by heat treatments/ or shear carried out under specific moisture conditions, followed by cooling phases during which part of the starch, mainly amylose, can create a three-dimensional network by strongly linking short starch chains by junction zones (Mestres et al., 1988). These modifications of starch can be induced during the technological process or, as an alternative, pre-gelatinized starches or starchy flours can be used as raw materials. In addition, other ingredients can be included in the formulation of GF pasta (GFP), improving its nutritional value. Nowadays, the most used ingredients in GFP production are rice and corn flours (Arendt et al., 2008), flours from pseudo-cereals (Caperuto et al., 2001; Chillo et al., 2008), starches of different origin (Huang et al., 2001), dairy products and vegetable proteins (Wang et al., 1999). In some cases, also low amounts of emulsifiers (Charutigon et al., 2008; Chillo et al., 2008) and hydrocolloids (Singh et al., 2004) are added.

Research on gluten free pasta using teff and millet solely is limited. Anna et al., 2012 used fresh egg, teff and oat flour to produce high protein pasta. Teff, quinoa and buckwheat pasta was nutritionally superior to regular wheat pasta. Quinoa and teff are characterized by high protein, fiber content and are high in calcium, magnesium and iron (Hager et al., 2011). Similarly, blend of proso millet and wheat flour yielded into high quality pasta (Sudha et al., 2015). Noodles made with 20% millet flour were of superior quality over wheat pasta but as the proso millet flour added to 40 and 60%, the texture and taste of noodles shown adverse results (Lorenz et al., 1980. Most studies have been carried out on the production of gluten-free products, such as breads, pasta, biscuits and beer, and the level of dietary fiber was increased by using different sources, such as inulin (Gibson and Roberfroid, 1995), corn starch (Gambus and Sabat, 2002), quinoa (Taylor and Parker, 2002), and amaranth (Tosi et al.,1996). But limited studies have been carried out where pasta is produced majorly either from teff or millet. This study is designed to use extrusion technology with two different grains with corn starch and mono-glycerides as texture enhancers for GF pasta. The objectives of this are to understand the processing nature of both teff and millet as base material and measure the physicochemical structure of cooked pasta.

Materials and Methods

Raw Materials

Durum wheat semolina was purchased from local stores, maker Ziyad Brand (12.74% moisture). Ivory teff flour of mean particle size 124 μm with 83% purity (9.94% moisture) and teff grain (9.52% moisture) were obtained from The Maskal Teff company. Yellow amber proso millets grain (11.56% moisture) was obtained from Hilary's Eat Well Company. Proso millets grains were ground to flour of particle size 115 μm in a table top lab scale roller mill, make Ross Mill. Native (10.38% moisture) and high amylose corn starch (12.01% moisture) were donated by Ingredion. Distilled mono-glycerides Dimodan HS-KA was purchase from Danisco. Barilla rotini durum wheat pasta and commercial rice based pasta was used as a control for quality comparison.

Proximate Analysis of Raw Materials

The proximate composition of raw ingredients was determined using standard methods. This included determination of moisture (135°C for 2h; AACC 44-19), crude protein (based on nitrogen by combustion, 6.25X; AOAC 920.176), crude fat (petroleum ether extract method; AOCS Ba 3-38), ash (600°C for 2h; AOAC 942.05), crude fiber (AOAC 962.09); and total starch (glucoamylase method; AOAC 979.10). Starch, protein, fat, ash and crude fiber contents were reported on dry basis percentage (%db) from replicates. Total carbohydrates were calculated by the difference method (Merrill and Watt, 1973).

Experimental Design

The experimental design includes usage of two grains teff and millet, with coarse and fine particle size of feed material. The treatments were formulated with three different concentrations (0%, 10% and 20%) of native and high amylose corn starch and two levels of mono-glycerides (0.5% and 1%) to produce high-quality spaghetti shaped pasta. The upper and lower limits of these levels were selected based on preliminary trials conducted on handmade pasta press machine (results not shown). There were eleven treatments in each grain experiment design. The response of each ingredients was analyzed by fitting quadratic models to the data with least square regression to identify significant ($p < 0.05$) effects of the variations in ingredient levels on the responses.

Extrusion Process

Dry ingredients were premixed in a planetary mixer (Hobart make) and water was added. Total mixing time was 5 mins for all treatments. All mixed hydrated ingredients were transferred into sealed plastic zipper and were let to hydrate overnight in a refrigerator to achieve uniform water distribution. The target moisture was 31% as is basis. Fresh pasta was produced using a lab scale co-rotating twin screw (American Leistritz, Somerville, NJ) with L/D ratio of 30:1, screw diameter of 18 mm equipped with

a spaghetti nozzle/ die (3.2 mm diameter), Figure 2.7. Raw materials blends were metered into the extruder with a twin screw volumetric feeder (K-Tron, Model K2VT20, North America, Pitman, NJ, USA). The feed rate was calibrated for each treatment independently and was kept between (0.85 to 0.95 kg/h). The screw speed was kept constant at 250 RPM and barrel temperatures were between 50- 85°C. Once out from die the product was cut into long strands (25 cm) using stainless-steel kitchen knife and packed into zipper pack plastic bags. The product was immediately stored in freezer to prevent starch retro-gradation. Drying was carried out at 70°C in hot air oven, relative humidity conditions were maintained by placing a bowl full of water inside oven. A hobo-meter was placed inside hot air oven to monitor humidity levels.

Cooking Process

Optimum cooking time for wheat pasta was the time required for the opaque central core of noodle to disappear when squeezed gently between two glass transparent plates, AACC Approved Method 66–50 (AACC, 2000). Similar scientific approach was used for teff and millet. An optimal cooking time for wheat pasta was 44 mins, 36 mins for teff and 25 mins for millet. Commercial wheat and rice pasta control was cooked as per cooking instructions which was 12 mins. The cooking time for all pasta was kept constant for the determination of cooking loss, water absorption/ weight gain, firmness and springiness.

Cooking Loss

Dry matter losses during cooking were determined by AACC Approved Method 66–50 (AACC, 2000). Pasta samples (25 gm) were cooked to optimum time in 300 mL of distilled water in a glass beaker, rinsed in a stream of cold water for 30 seconds and drained. Rinsed water was collected, and the volume made to 500 mL. The beakers carrying liquid were evaporated to dryness in air oven at 100±1°C. Drying time was approximately 24h but may vary with oven capacity, load, etc. Once completely dry the

beakers were taken out from oven and cooled in desiccator and weighed. Duplicate samples were carried out for precise data. The formula used to calculate cooking loss is given below.

$$\text{Cooking loss} = \frac{\text{Dried weight of residue}}{\text{Weight of sample}} \times 100$$

Water Absorption

Water absorption or hydration is the amount of water retained by the cereal products. It is related to functionality parameters such as cooking time and texture following cooking. Water uptake in pasta is one the most important parameter post cooking that not only related to weight gain but also affects the taste. It is measured during the cooking loss analysis. The drained pasta weighed was measured for water absorption. All the samples were subjected to duplicate for precise results. Below mentioned was the formula used to calculate water absorption/uptake.

$$\text{Water absorption \%} = \frac{(\text{Cooked product weight} - \text{Dry product weight})}{\text{Dry product weight}} \times 100$$

Thermal Analysis-Differential Scanning Calorimetry

To understand the physical transformation of starch and protein known as starch gelatinization and protein denaturation, calorimetric measurements were carried out for each raw material blends and extruded products. A differential scanning calorimeter (DSC) instrument Q100 DSC (TA Instruments, New Castle, DE, USA) was used for analysis. A sample of 8-10 mg was weighed into large volume stainless steel DSC pans (Part no.03190029, Perkin Elmer Health Sciences Inc., Shelton, CT, USA). Distilled water was added to the sample in the pan to obtain a solid to water ratio of 1:2 (Stevens and Elton, 1971; Zhu et al., 2010). The pans were hermetically sealed and the samples allowed to equilibrate overnight. The instrument was calibrated using indium as

reference material. An empty sealed pan was used as reference for all experiments. The program consisted several steps which include both heating and cooling steps is as follows. Equilibrate at 10°C, heating the pans from 10°C to 140°C at the rate of 10°C/min, mark end of cycle, cooling down the sample from 140°C to 10°C at the rate of 25°C/min, mark the end of cycle with nitrogen gas flow rate of 50mL/min. The samples were again rescanned with heating from 10°C to 140°C at the rate of 10°C as the final phase of the test. DSC datum for each gelatinization and denaturation endotherm was analyzed for transition temperatures, onset (T_o), peak (T_p), and endpoint or completion (T_c) and the enthalpy (ΔH) using TA Instruments Universal Analysis Software (version 5.4.0). All the reported data was subjected to duplicates.

Starch gelatinization (%) was calculated by comparing the enthalpy transition difference in starches between raw and extruded binary blends. Calculations were made using the equation below:

$$\text{Starch gelatinization \%} = \frac{\Delta H_{\text{raw}} - \Delta H_{\text{extruded}}}{\Delta H_{\text{raw}}} \times 100$$

Where, ΔH_{raw} = enthalpy of raw binary blend,

$\Delta H_{\text{extruded}}$ = enthalpy of extruded binary blend

Total cook (%) was calculated as a ratio of the total enthalpic transition difference which includes the transition enthalpies for starch and protein fractions of the binary blends. It is represented as below:

$$\text{Total cook \%} = \frac{\Delta HT_{\text{raw}} - \Delta HT_{\text{extruded}}}{\Delta HT_{\text{raw}}} \times 100$$

Where, ΔHT_{raw} = Total enthalpy of transition of raw binary blend,

$\Delta HT_{\text{extruded}}$ = Total enthalpy of transition of extruded binary blend

Pasting Properties

The Rapid Visco Analyzer (RVA) provides a measure of how cooked a sample is by re-cooking under relatively low shear and in excess water and measuring the pasting viscosity throughout the test. Pasting properties of each flour and blends were examined using RVA. (RVA 4, Newport Scientific Pvt. Ltd., Warriewood, NSW, Australia). The RVA was interfaced with a computer equipped with the software – ThermoLine for Windows (version 3.15.2.298) for controlling the test and analyzing the results. The sample size was 3.5 to 4.0 g and the amount of water added was 24.0 to 25.0 ml (corrected for 14% moisture basis). Pasting properties were determined after running the samples on standard AACC profile (AACC 76-21.01, 1999) with a run time of 13 minutes. The temperature range was fixed between the set points; rate of heating and cooling was 12°C per min. The paddle was set at 960 rpm for first 10 secs then 160 rpm for the remaining test time. Peak viscosity (PV), pasting temperature (PTc), trough viscosity (TV), breakdown (BD), final viscosity (FV) and setback (SB) were recorded. The viscosity measurement was done in cP (centipoise) units. All measurements were performed in duplicate for both raw material and extruded products.

Texture Profile Analysis

The texture properties of the pasta after cooking are extremely important macroscopic chemical-physical properties for assessing the quality of the pasta, given the fact that they represent some of the characteristics that the consumer is more attentive now during consumption. A texture profile analysis (TPA) of pasta was conducted to determine firmness and springiness of cooked pasta and brittleness of raw pasta by using the method of Voisey et al. (1978) and Tang et al. (1999) with modification. Pasta texture was evaluated using a TA.XT plus texture analyzer system (Stable Micro Systems, Surrey, UK). The flexural strength or breaking strength/stress of uncooked (rigid) samples was determined by performing a three-point bend test. The uncooked pasta samples were cut to 5 cm length. The fixture was placed to supports the sample across a span to hold the sample horizontally across the test probe (TA46 blind edge). A force is applied to the center of the sample (which is also central to the supports) and

the breaking stress is determined. The breaking strength (force per unit width) or breaking stress (force per unit area) of the sample is taken as the maximum strength or stress value of the curve. The distance to break gives an indication of the brittleness of the sample as this shows how far a sample can be deformed before fracture. The gradient of the slope indicates sample toughness; the higher the gradient, the tougher the sample. Test parameters were set as follows: test mode= compression; pretest speed= 1.0 mm/sec; test speed= 1.0 mm; post-test speed=10.0 mm/sec; target mode = distance; distance= 15.0 mm; trigger type=auto-force; trigger force= 5.0 gm.

Firmness of the cooked noodles was measured by AACC Approved Method 66–50 (AACC, 1999). Firmness is defined as the maximum force at the first compression. The test is a simulation of the action of jaw by compressing the bite size of food two times. The resulting force–time curve is used to extract number of textural parameters. These are primary parameters (hardness, springiness and adhesiveness). The parameters for the test were set as follows: test mode= compression; pretest speed= 1.0 mm/sec; test speed= 1.0 mm; post-test speed=1.0 mm/sec; target mode = strain; strain= 85%; count =2; trigger type=auto-force; trigger force= 5.0 gm. A 2" diameter and 20 mm tall cylindrical aluminum probe was used for test. To manage the standard deviations, results were obtained after taking the average of 25 measurements.

Statistical Analysis

All the results were analyzed using analysis of variance (ANOVA) with general linear model procedure (SAS version 9.1, SAS Institute, Cary, North Carolina, USA). When significant effects ($p \leq 0.05$) were indicated by ANOVA, Tukey pairwise comparisons were conducted to distinguish which treatments differed significantly ($p \leq 0.05$). Pearson Correlations was used to establish correlation values.

Results and Discussion

Proximate Analysis of Raw Materials

The proximate analysis of raw ram material is represented in Table 2.1. Millet (70.2%) was the highest in starch content followed by semolina (65.7%) and teff (64.8%). High starch content is most desirable for precooked pasta to form a cooked product matrix. Similarly, millet was also rich source fat (4.1%), followed by teff (2.0%) and semolina (1.1%). As expected semolina was highest in protein content (13.8%) followed by teff (11.7%) and millet (10.4%). Teff had the highest crude fiber content (1.8%) with millet stood at second place (0.8%). Hypothetically with high starch, low fiber and moderate protein content millet should have resulted into better precooked pasta but our results were contrary. The possible reason could be the domination of fat and added lipid (MG) into the millet flour may have impaired extrusion cooking resulted into poor quality product. The second reason could be the nature of millet starch granules which resulted into poor binding. Although, there is limited amount of research work done in these grains for affirmative reasoning.

Pasta Production

The pasta was produced at 28.58% to 31.31% feed moisture. The moisture data for raw blends, post extruder and post drying is shown in Appendix B, Table 1. The process moisture loss for teff pasta ranged from 3.6% to 17.9% and for millet it ranged from 5.0% to 16.5%. The process moisture losses in millet were lower over teff because of low energy input. The rich fat content of millets and added mono-glycerides into blends acted as lipid which lowered process energy input. The motor load variations for teff and millet blends as represented in Table 2.2. The increase in mono-glycerides concentration of blends decreased motor load. The net motor load ranged from 4.8% to 11.3% in millet processing and 10.3% to 12.3% in teff processing. Lipids act as a lubricant inside extruder barrel, lowers the energy input which led to low shear. The lubricating effect of lipid reduces the friction between feed material and screw surfaces (Guy, 2001; Gour and Gautam, 2003). For coarse grains moisture loss was higher in

millet than teff. It was observed that both millet and teff coarse grains did not processed completely inside extruder barrel. In overall teff pasta has required higher energy inputs over millet during processing.

The moisture content of dried pasta is represented in Appendix B, Table 1. During drying the moisture losses of millet pasta ranged from 47.8% to 82.1% and from 52.2% to 59.5% for teff pasta (Figure 2.2). Teff processed at higher energy achieved higher degree of gelatinization over millet (Table 2.4). The higher degree of cooking in teff pasta has formed a strong starch matrix which may have prevented the water evaporation during drying. Whereas, low degree of gelatinization in millet pasta led to lose starch matrix resulted into higher water evaporation. Bruneel et al. (2010), also reported higher moisture loss during wheat pasta drying at lower degree of gelatinization and vice versa.

Cooking Loss

The cooking losses of teff and millet pasta are shown in Table 2.3. The optimal cook time of teff and millet was different. The difference between optimal cooking times may be attributed to the difference in the gelatinization temperatures of teff and millet starch (Singh and Singh, 2002). No information is currently available about the effect of corn starch and mono-glycerides addition on optimum cook time and other characteristics for teff and millet pasta.

The cooked pasta characteristics are the results of several phenomena occurring during cooking such as hydration, starch gelatinization and interaction with non-starchy matrices. Cooking losses below 10% of pasta mass indicates good quality of precooked pasta (Kim et al., 1996; Wang et al., 1999). In general, cooking losses of teff and millet pasta were comparable to those of lab control wheat pasta. Among teff blends, the cooking losses were highest for teff formulated with (10%) native corn starch and 1% MG; cooking losses of six treatments out of eight were lower than 10%, indicating their good quality (Table 2.3). Overall, the addition of native corn starch and MG controlled mean cooking losses of both teff and millet pasta (Figure 2.3). However, the addition of

high amylose starch resulted in higher cooking loss. Twenty percent addition of native corn starch reduced cooking losses by 23% with 0.5% MG addition and 19.5% at 1% MG addition (Appendix A; Figure 1 and 2).

High starch degradation due to excessive cooking can also be a major factor influencing high cooking losses observed during boiling (Abecassis et al., 1994; Kruger et al., 1996). Regression analysis showed that 21% of variation in cooking losses can be explained by the degree of starch gelatinization ($R^2= 0.21$) in teff pasta. The cooking losses were higher for high level corn starch formulations (Appendix A; Figure 3 and 4). Precooked pasta formulated with high amylose starch resulted in higher mean solid losses (Figure 2.4) due to leaching of amylose during cooking (Kim et al., 1996).

The increase of (0.5%) mono-glycerides concentration increased mean cooking loss with both native and high amylose corn starch (Figure 2.3), and due to its effect on starch gelatinization. The addition of mono-glycerides produce lubrication between feed particles and screw surface which lowers the barrel pressure and shear. The lower energy input inhibits swelling of starch granules in water, and reduces starch gelatinization (Table 2.4). The lower motor load torque (Table 2.2) and low degree of gelatinization for teff pasta at higher MG levels are represented in Table 2.4. The lower degree of cooking results into higher cooking loss (Resmini et al., 1979). Chanpen et al., 2007 also found increased cooking loss when emulsifier concentration was increased to 1.0 and 1.5 g/100 g blend.

In millets, cooking losses were higher than 10% indicates the poor quality of millet pasta (Table 2.3). Wang et al. (1999) reported 20.5% cooking loss for GF pasta produced from pea flour at 110°C, and up to 48.2% for products extruded at low temperature. The induction of non-gluten flours in some pasta affects the quality attributes (e.g. higher cooking loss, lower breaking energy), that can contribute to the nature of non-gluten protein and insoluble fiber which weakens the overall structure of pasta (Petitot et al., 2010; Petitot et al., 2010). However, the addition of 20% native corn starch and 1% mono-glycerides have reduced cooking loss in seven treatments out of eight but still the losses were higher than 10% (Appendix A; Figure 2). The possible reason for higher

cooking losses may be the formation of amylose-lipid and amylose-mono-glycerides helical complexes which impedes degree of gelatinization (Table 2.5). Fifty seven percent of cooking losses variation for millet was due to degree of gelatinization ($R^2=0.57$). The lower energy inputs decreased the degree of starch gelatinization which resulted in higher cooking loss (Table 2.5). Overall teff pasta was of better quality with lower cooking losses. The cooking loss for commercial wheat pasta was $3.47\pm 0.3\%$ and for rice pasta it was $6.12\pm 0.1\%$.

Water Absorption

The weight of a cooked pasta is an indicator of water uptake and corresponds to a several macroscopic events involving a complex molecular modification of starch and proteins, mainly hydration (Sozer et al., 2007). Swelling of pasta occurs during cooking and water uptake shows how well pasta responds to cooking. Table 2.3 represents water absorption of all pasta types. Water absorption was significantly affected by the formulations; for teff the mean value was 164.4% and 159.1% for millet, for commercial wheat pasta it was 227% and 168% for rice pasta. The half percent increase of MG in formulations increased mean water absorption significantly for both teff and millet (Figure 2.5). The water absorption of finished teff pasta was highly correlated with moisture content of dried pasta ($R^2= 0.83$) and MG levels ($R^2= 0.74$) of formulations. Similarly, for millet pasta water absorption correlation value with dried moisture content was ($R^2= 0.33$) and for MG levels ($R^2= 0.32$).

Water absorption is a measure of intact starch granules after extrusion processing, and it can be used as an index of starch gelatinization (Ding et al., 2005). Water absorption of precooked pasta is mainly dependent on the intensity of baro-thermal treatment and degree of starch gelatinization during processing (Wojtowicz, 2005). Pasta processed at higher motor load had high degree of starch gelatinization and absorbed more water. Hence, water absorption values are related to degree of starch gelatinization. The water absorption of teff pasta ranged from 133.3% to 196.3%; the highest value was shown for the formulation with 20% native corn starch and 1% MG; for millets, it ranged from

140.7% to 174.5%; the highest was with 20% high amylose corn starch and 1%MG.

Twenty-nine percent variation in water absorption can be explain by degree of gelatinization ($R^2= 0.29$) in teff and twenty-five percent in millet pasta ($R^2= 0.25$). The combination of twenty percent of corn starch and one percent of mono-glycerides had significant influence on water absorption (Appendix A; Figure 10, 12 and 14). The experiment involved mono-glycerides which inhibits swelling of starch granules during gelatinization by forming water-insoluble complexes with amylose. The addition of one percent mono-glycerides lowered the energy input and reduced starch gelatinization. The poorly cooked pasta with weak structure formation absorbed less water (Wojtowicz, 2005).

In both teff and millets the weight of precooked pasta enriched with 20% high amylose starch and 1% MG was highest (Appendix A; Figure 12 and 16). It is known that amylose has higher water binding capacity than native starch (Zhiqiang et al., 1999). Hence, high values of water absorption can be attributed to high levels of amylose in the formulation. However, overall the native corn starch formulations yielded high mean water absorption over high amylose starch formulations (Figure 2.6). Teff had higher degree of gelatinization therefor teff pasta absorbed more water than millet pasta. Therefore, teff has high water absorption capacity, which relates to higher swelling degree of gel phase of teff starches. The small and uniform size of teff starch granules provides larger surface area for higher water penetration this could also be the possible reason for high water absorption (Bultosa, 2004; Bultosa et al., 2002).

Thermal Analysis-Differential Scanning Calorimetry

Starch gelatinization and melting of crystalline structure is an important phenomenon occurs in several food processing operations because it delivers unique textural and structural characteristics to the products. The native starch granule is partially crystalline polymer which losses it's crystalline and molecular order during gelatinization. The gelatinization temperature is a characteristic of the starch type and depends on the glass transition of the amorphous region (Eerlingen and Delcour, 1995). It is pertinent to

acquire knowledge of the kinetics of starch gelatinization and melting in cooking of pastas (Spigno and Faveri, 2004). Starch is a major component of structure and firmness in precooked pasta, and is influenced by gelatinized starch properties.

Table 2.4 and 2.5 shows the mean values for DSC gelatinization (cooking) of semolina, teff and millet precooked samples. Teff pasta onset and peak temperatures of gelatinization were found between 81.6°C and 85.9°C; for millet pasta, it was between 80.5°C and 85.3°C. The analysis of peak temperatures did not make so much difference in teff but for millet samples peak temperature values were significantly different. The range for gelatinization temperature was 11.35°C for teff and 12.26°C for millet. Teff starch gelatinization temperature range measured on DSC by Bultosa et al. (2003) was 64°C-80°C which is similar to results shown in this study. The onset and peak temperature values for semolina were lower than teff and millet.

The degree of starch gelatinization for teff ranged from 91.1% to 99.9%. The gelatinization values for teff pasta variants were significantly different ($p < 0.05$). It has been noticed that lipid rich millet blends had lower values of gelatinization. Similarly, total cook of the millet pasta blends after considering total enthalpy (starch gelatinization and protein denaturation) ranged from 68.5% to 95.5% (Table 2.5). The gelatinization values for millet pasta variants were significantly different ($p < 0.05$). In general, the percentage of starch in the blends affects the gelatinization percentage. The starch content of teff flour was 64.8% and millet flour was 70.2% (Table 2.1). Thus, higher starch content of millets could have contributed towards lower transition enthalpy during the starch gelatinization process but high inherent fat might have formed amylose –lipid complex during thermal transition process and thereby lowering gelatinization when compared to teff. Even, formulations with higher MG concentrations led to high transition enthalpy resulted into lower degree gelatinization. The lower energy transfer to blends during processing because lipids provides a lubricating effect (Feng and Lee, 2014), that reduces starch cooking in extruder.

Pasting Properties

Rapid Visco Analyzer (RVA) was used for offline control to measure relative starch degradation (Ryu et al., 1993). Pasting properties of starch are the phenomena involving granular cooking, swelling, and total disruption of granules (Atwell et al., 1988). It has been used to quantify cold-swelling of 'cooked' component, 'raw' component that paste's during test and overall viscosity that indicates degree of starch dextrinization. Pasting properties of raw teff blends are shown in Table 2.6, and raw millet samples are represented in Table 2.8. The peak viscosity values of control wheat were highest 2357 ± 22.6 , followed by teff 1985 ± 30 and millet was lowest 1637 ± 50 . Peak viscosity values for both teff and millet blends were significantly different ($p < 0.05$). The inclusion of native corn starch increased peak viscosity for both teff and millets, whereas addition of high amylose glucose decreased peak viscosity (Appendix A; Figure 18 and 19). Raw teff flour blends have shown highest mean pasting temperature $78 \pm 0.1^\circ\text{C}$, millet was $73.3 \pm 0.1^\circ\text{C}$ and wheat was $69 \pm 0.1^\circ\text{C}$. Pasting temperatures of both raw teff and millets were significantly different ($p < 0.05$) for each treatment. The breakdown viscosity value of control wheat was highest, followed by millet and teff. Millet has shown lower peak viscosity, lower peak temperature and higher breakdown viscosity over teff flour. High lipid concentration of millets facilitates slippage action which impedes starch granule cooking and swelling. The restricted swelling of starch granules impact viscoelastic properties adversely and results into low pasting and breakdown viscosity values (Raphaelides and Georgiadis, 2006). Millet pasting property results agree with Qingjie et al. (2014). RVA results indicated that teff is more resistant to shear and require higher energy and temperature to reach peak viscosity than millets. Bultosa et al. (2002) also reported that teff starch is more resistant to shear than maize starch. The findings of this study are also backed by motor load data represented in Table 2.2. Teff required higher specific mechanical higher to process over millets at same conditions.

Pasting curves of the extruded samples were significantly different from raw samples. These changes in the pasting characteristics indicate that starch molecules had some degree of re-association and interaction with non-starch ingredients during high thermal

and shear processing. It also suggested that shear-stabilization of the starch granule was provided by thermal processing, which was like modified starches obtained by chemical cross-linking. Pasting properties of extruded precooked pasta for teff and millet are shown in Table 2.7 and 2.9. Peak viscosity values of precooked pasta treatments were lower than raw material blends which are the indicator of starch granule cooking and disintegration during extrusion processing. Appendix A; Figure 20, 21 and 21, are representing trends of precooked teff samples. Lab control wheat showed highest mean peak viscosity figures for cooked products, followed by teff and millets pasta. Peak viscosity values of extruded treatments were significantly different ($p < 0.05$). Increase of 0.5% mono-glycerides increased peak viscosity among all teff pasta treatments. Addition of native corn starch has increased peak viscosity of cooked millet pasta, and high amylose has reduced PV (Appendix A; Figure 26, 27 and 28). No such trend was observed in teff pasta. The mean pasting temperature was highest for teff 76°C, millet was 60°C, and lab control wheat was 50.5°C. Pasting temperatures were significantly different ($p < 0.05$). Millet has shown lower breakdown viscosity values which may be attributed to low cooking of millets. The differences of pasting characteristics among teff and millets may be attributed to the difference in the starch granule structure (Li et al., 2008; Lim et al., 2003), which need further investigations in the future.

Textural Properties

Firmness and springiness are the two most important textural parameters for cooked pasta quality. The mean firmness value (10.4kg) of cooked teff pasta samples were comparable to control semolina pasta; highest (14kg) was with 10% high amylose starch and 0.5% MG; and lowest (8.5kg) was with 10% native starch and 1% MG. Firmness of cooked teff pasta with 0.5% MG was 10.4kg, and 10.6kg for semolina, Table 2.10. Firmness of a cooked pasta or spaghetti is mainly governed by post cooking characteristics of protein and bran of pasta (Matsuo and Irvine, 1970). Data analysis showed a positive linear coloration ($R^2 = 0.62$) between gelatinization percentage and firmness. The possible reason could be higher degree of cooking that might have

increased hardness of cooked teff pasta. No literature is available on firmness behavior of teff starch and protein post cooking. The firmness values of teff pasta formulated corn starch and MG were highly correlated with degree of gelatinization. A trend of increase in firmness with increase in gelatinization degree has been noticed, the linear correlation between firmness and degree of gelatinization was ($R^2= 0.61$). Similar trend of increase in firmness of cooked pasta with higher degree of cooking were reported by Soh et al. (2006). Pasta formulated with high levels of MG had lower firmness due to less cooking. The addition of emulsifier such as mono-glycerides significantly affected the degree of gelatinization in high shear process (Chanpen et al., 2007).

Millet pasta was less firm than teff, Table 2.10. The mean firmness value of millet pasta samples was 7.7 kg; highest (8.7kg) was for 10% native starch and 1% MG; lowest (5.5kg) was for 20% native starch and 0.5% MG. It can be concluded that firmness of cooked millet pasta is lower than teff. The correlation values of firmness with gelatinization was ($R^2=-0.24$); and with native corn starch levels was ($R^2= -0.58$). Firmness values for cooked commercial wheat pasta were 5.4 kg and 9.5kg was for commercial rice pasta.

There was a tendency for firmness to increase as amylose starch content increased in both teff and millet pasta, but only high amylose samples were significantly higher in firmness. Dexter and Matsuo (1979) used blends of semolina starch and amylo-maize-7 starch (a high amylose starch, $\approx 52\%$) and found cooked pasta became firmer as proportion of amylo-maize starch was increased. In juxtapose, decreasing amylose below normal levels causes a decrease in pasta firmness (Gianibelli et al., 2005; Vignaux et al., 2005). In high-amylose starches granules are more tightly packed and, on swelling, have more resistance to rupture and deformation. Extrusion conditions also influenced cooking qualities of pasta. In particular, positive effects of extrusion cooking reduced cooking loss and increased cooked product firmness. This behavior could be related to formation of a polysaccharide network involving starch and non-starch polysaccharide molecules. According to Gualberto et al., (1997), high physical stress during extrusion-cooking reduces amount of insoluble fiber. Therefore, formation of a

higher amount of soluble fiber after extrusion-cooking could enable a strengthening of pasta structure, as supported by Tudorica et al. (2002) in durum wheat pasta.

Springiness was defined as distance at which a deformed sample went back to its non-deformed condition after deforming force is removed during second compression. No significant difference was observed among springiness of cooked pasta made from pasta fortified with corn starch and MG at all levels (Table 2.10). Addition of MG into the formulation reduced springiness of cooked pasta but reduction was not significant. Springiness values of cooked teff pasta ranged from 1.07 to 1.01; highest was for teff with 0.5% MG and for cooked millet pasta it ranged from 1.03 to 1.00. Increase in cooking loss observed in pasta was likely due to MG addition, responsible for low starch cooking. At same time, inclusion of MG partially reduced firmness and springiness found in teff and millet pasta.

Three-point bend test

The force required to break the uncooked pasta from three points was presented in Table 2.10. The control samples (semolina) required the highest force to break, followed by teff and lowest was for millet pasta. Mean values of force for teff were 2.12kg; mean value for millet was 0.35kg and for semolina it was 5.38kg. Among teff treatments highest forces (3.32kg) required was for 10% high amylose starch and 0.5% MG, lowest (0.94kg) was for 10% native starch and 1% MG. A negative correlation ($R^2=-0.96$) for force values with MG levels; a positive correlation ($R^2=0.82$) with moisture content of dried pasta, and ($R^2=0.33$) with degree of gelatinization was found in teff treatments. Mean force required to break the uncooked millet pasta was 0.35kg; highest (1.39kg) for 20% native starch and 0.5% MG; lowest (0.11kg) for 10% native starch and 0.5%MG. Correlation values for force with MG levels was ($R^2=0.14$), was ($R^2=0.32$) with dried pasta moisture content, and was ($R^2=0.14$) with degree of gelatinization. Firmness values for commercial wheat pasta were 1.10 ± 0.41 kg and 2.21 ± 0.32 kg for commercial rice pasta.

Conclusions

The present study supported the hypothesis that non-traditional grains flours are useful ingredients for producing optimum quality pasta, and for nutritional improvement of gluten free products. Using native corn starch and mono-glycerides as a functional ingredient resulted in a good quality product with lower cooking loss, high water absorption, higher degree of and gelatinization. The cooked teff pasta was comparable to control but millet pasta was of inferior quality. The consumption of gluten free spaghetti formulated with teff and millet flour can have positive implications for human health, due to their increased nutritional quality and favorable food choice to people suffering from celiac diseases. The incorporation of non-traditional grain into human food chain can open a wide range of solution to economics, climate change, mitigating global hunger and malnutrition.

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Table 2.1: The proximate analysis of raw materials.

Treatments	Crude Protein (%)	Crude Fiber (%)	Fat %	Ash %	Total starch %
Semolina	13.8±0.0	<0.2±0.0	1.1±0.0	0.7±0.0	65.7±0.4
Teff flour	11.7±0.1	1.8±0.2	2.0±0.0	1.9±0.1	64.8±0.4
Millet flour	10.4±0.5	0.8±0.2	4.1±0.1	1.2±0.0	70.2±0.5

Table 2.2: Net motor load of extruder in teff and millet pasta processing.

Treatments	Motor Load (%)	
	Teff	Millet
Control	13.0	12.0
MG(0.5)	10.5	7.0
CS28AM (10)+MG(0.5)	10.8	6.0
CS28AM (10)+MG(1)	10.3	4.8
CS28AM (20)+MG(0.5)	11.5	11.3
CS28AM (20)+MG(1)	11.0	6.5
CS55AM(10)+MG(0.5)	12.3	6.3
CS55AM(10)+MG(1)	11.0	4.8
CS55AM(20)+MG(0.5)	12.3	10.5
CS55AM(20)+MG(1)	10.3	6.0
Coarse Grain	7.3	9.5

Legends: Control wheat- durum wheat semolina pasta, CS28AM-28% amylose corn starch, CS55AM-55% amylose corn starch, MG(0.5)- 0.5% mono-glycerides, MG(01)- 1.0% mono-glycerides

Table 2.3: Cooking loss and water absorption of teff and millet pasta.

Treatments	Water absorption (%)		Cooking loss (%)	
	Teff	Millet	Teff	Millet
Control	148.4±1.2 ^{hfg}		07.9±0.1 ^e	
MG (0.5)	143.6±2.4 ^{fg}	146.0±4.7 ^{bc}	11.6±0.1 ^c	15.6±0.6 ^{bc}
CS28AM (10)+MG (0.5)	162.4±1.2 ^{edf}	171.9±2.1 ^a	10.3±0.2 ^{dc}	19.1±2.2 ^{ba}
CS28AM (10)+MG (1)	189.6±0.6 ^{cb}	164.7±6.5 ^{ba}	14.1±0.9 ^b	17.8±0.4 ^{bac}
CS28AM (20)+MG (0.5)	147.6±2.7 ^{hgf}	152.5±1.3 ^{bac}	08.9±0.4 ^{de}	10.1±0.6 ^{ed}
CS28AM (20)+MG (1)	196.3±0.0 ^b	167.2±0.3 ^{ba}	09.7±1. ^{de}	09.8±0.1 ^{ed}
CS55AM (10)+MG(0.5)	133.3±1.0 ^h	157.0± 2.4 ^{bac}	09.4±0.1 ^{de}	16.4±1.0 ^{bac}
CS55AM (10)+MG(1)	171.01±3.6 ^{ed}	156.9±2.6 ^{bac}	11.5±0.4 ^c	20.6±1.4 ^a
CS55AM (20)+MG(0.5)	158.4± 0.8 ^{egf}	140.7±0.9 ^c	10.4±0.5 ^{dc}	14.5±1.3 ^c
CS55AM (20)+MG(1)	177.3±11.8 ^{cd}	174.5±2.0 ^a	10.4±0.4 ^{dc}	14.0±1.4 ^{dc}
Coarse Grain	224.4±6.1 ^a	160.4±16.4 ^{bc}	26.8±0.2 ^a	18.1±0.2 ^e

Legends: Control wheat- durum wheat semolina pasta, CS28AM-28% amylose corn starch, CS55AM-55% amylose corn starch, MG(0.5)- 0.5% mono-glycerides, MG(01)- 1.0% mono-glycerides.

Table 2.4: Thermal characteristics of teff precooked pasta samples: onset (T_o), peak (T_p), completion (T_c), gelatinization enthalpies (ΔH) and degree of starch gelatinization (Δg).

Treatments	T_o (°C)	T_p (°C)	T_c (°C)	ΔH (J/g)	Δg (%)
Control	55.5±0.11 ^d	60.3±0.25 ^c	70.7±0.57 ^b	0.56±0.14 ^{bc}	94.3±1.91 ^{dc}
MG (0.5)	80.3±0.13 ^{bc}	83.6±0.26 ^{ba}	91.5±0.03 ^a	0.60±0.07 ^{ba}	93.7±0.76 ^{dc}
CS28AM (10)+MG (0.5)	80.6±0.02 ^{bac}	83.7±0.16 ^{ba}	91.2±0.11 ^a	0.39±0.01 ^{bcd}	95.6±0.65 ^{bac}
CS28AM (10)+MG (1)	79.8±0.40 ^c	83.6±0.92 ^{ba}	91.1±1.14 ^a	0.83±0.08 ^a	91.2±0.10 ^d
CS28AM (20)+MG (0.5)	81.0±0.08 ^{ba}	85.3±0.93 ^b	92.4±1.42 ^a	0.31±0.05 ^{ed}	96.2±0.48 ^{bac}
CS28AM (20)+MG (1)	80.7±0.21 ^{bc}	83.0±0.74 ^b	90.3±1.73 ^a	0.60±0.03 ^b	91.1±1.87 ^d
CS55AM(10)+MG(0.5)	81.4±0.16 ^a	86.0± 0.04 ^a	91.5±0.12 ^a	0.07±0.02 ^f	99.1±0.34 ^a
CS55AM(10)+MG(1)	80.1±0.19 ^{bac}	84.6±0.09 ^{ba}	92.0±0.25 ^a	0.40±0.02 ^{bcd}	93.9±0.88 ^{dc}
CS55AM(20)+MG(0.5)	80.1± 0.64 ^{ba}	85.3±0.78 ^{ba}	91.1±0.68 ^a	0.07±0.01 ^f	99.1±0.12 ^a
CS55AM(20)+MG(1)	81.0±0.10 ^{ba}	84.6±0.05 ^b	91.8±0.13 ^a	0.09±0.03 ^{fe}	98.7±0.55 ^{ba}
Coarse Grain	81.1±0.43 ^{ba}	85.7±1.45 ^{ba}	93.8±2.78 ^a	0.36±0.03 ^{cd}	95.3±0.45 ^{bc}

Legends: Control wheat- durum wheat semolina pasta, CS28AM-28% amylose corn starch, CS55AM-55% amylose corn starch, MG(0.5)- 0.5% mono-glycerides, MG(01)- 1.0% mono-glycerides. Enthalpy ΔH (J/g) of raw flours; semolina $\approx 9.02 \pm 0.21$, sorghum flour $\approx 8.60 \pm 1.65$, teff flour $\approx 10.15 \pm 0.18$, millet flour $\approx 6.84 \pm 0.50$

Table 2.5: Thermal characteristics of millet precooked pasta samples: onset (T_o), peak (T_p), completion (T_c), gelatinization enthalpies (ΔH) and degree of starch gelatinization (Δg).

Treatments	T_o (°C)	T_p (°C)	T_c (°C)	ΔH (J/g)	Δg (%)
Control	57.7±1.68 _b	58.8±0.25 ^c	63.7±0.59 ^c	0.27±0.01 ^e	99.2±0.10 ^a
MG (0.5)	80.1±0.52 _a	84.0±0.85 ^{ba}	90.6±0.95 ^a	0.61±0.03 ^{dc}	92.4±0.81 ^{dc}
CS28AM (10)+MG (0.5)	77.6±0.54 _a	82.0±0.54 ^{ba}	90.4±0.63 ^a	2.09±0.12 ^a	71.9±0.89 ^{fe}
CS28AM (10)+MG (1)	78.1±0.04 _a	82.4±0.07 ^{ba}	89.3±0.82 ^a	2.3±0.12 ^a	68.5±3.53 _f
CS28AM (20)+MG (0.5)	80.0±1.12 _a	82.4±1.03 ^{ba}	90.7±0.60 ^a	0.58±0.04 ^{dc}	94.25±0.48 ^{bdac}
CS28AM (20)+MG (1)	80.6±0.03 _a	84.3±0.66 ^a	91.6±1.53 ^a	0.71±0.08 ^{dc}	92.1±0.98 ^{dc}
CS55AM(10)+MG(0.5)	79.5±0.32 _a	83.7± 0.73 ^{ba}	91.6±0.64 ^a	0.87±0.02 ^c	89.8±0.36 ^d
CS55AM(10)+MG(1)	78.8±0.08 _a	82.7±0.13 ^{ba}	90.3±1.28 ^a	1.76±0.00 ^b	77.6±0.53 ^e
CS55AM(20)+MG(0.5)	78.7± 0.64 ^a	82.2±0.57 ^{ba}	90.7±0.93 ^a	0.43±0.06 ^d	95.5±0.55 ^{bac}
CS55AM(20)+MG(1)	80.5±0.97 ^a	83.7±1.70 ^{ba}	91.6±1.15 ^a	0.51±0.15 ^d	93.3±2.64 ^b
Coarse Grain	78.8±0.42 _a	81.2±0.74 ^b	85.1±0.18 ^b	0.11±0.04 ^e	98.5±0.51 ^{ba}

Legends: Control wheat- durum wheat semolina pasta, CS28AM-28% amylose corn starch, CS55AM-55% amylose corn starch, MG(0.5)- 0.5% mono-glycerides, MG(01)- 1.0% mono-glycerides. Enthalpy ΔH (J/g) of raw flours; semolina $\approx 9.02 \pm 0.21$, sorghum flour $\approx 8.60 \pm 1.65$, teff flour $\approx 10.15 \pm 0.18$, millet flour $\approx 6.84 \pm 0.50$

Table 2.6: Pasting properties of raw teff flour blends formulated with corn starch and mono-glycerides.

Treatments	PT _c (°C)	PT _m (mins)	PV (cP)	TV (cP)	BD (cP)	SB (cP)	FV (cP)
Control	63.0±0.6 ^c	5.8±0.1 ^{ba}	2358±23 ^a	1643±49 ^a	715±47 ^a	1970±153 ^b	3613±194 ^{ba}
MG (0.5)	78.7±0.2 ^{ba}	6.0±0.0 ^c	1985±30 ^{de}	1600±30 ^c	385±00 ^b	1593±33 ^c	3193±63 ^c
CS28AM (10)+MG (0.5)	79.4±0.1 ^a	5.9±0.0 ^c	2220±71 ^{cd}	1721±57 ^{cb}	449±14 ^{ba}	1536±29 ^c	3256±28 ^c
CS28AM (10)+MG (1)	78.6±0.0 ^{ba}	6.1±0.0 ^{bc}	2116±146 ^{cde}	1702±141 ^{cb}	414±4 ^b	1646±112 ^c	3348±29 ^c
CS28AM (20)+MG (0.5)	77.2±0.1 ^{ba}	5.9±0.0 ^c	2506±28 ^b	1904±31 ^b	602±59 ^a	1470±43 ^c	3374±12 ^c
CS28AM (20)+MG (1)	78.1±1.1 ^{ba}	6.2±0.1 ^{bac}	2288±52 ^{cb}	1883±74 ^b	405±21 ^b	2092±106 ^b	3975±180 ^b
CS55AM(10)+MG(0.5)	78.1±1.0 ^{ba}	6.2± 0.0 ^{bac}	1538±25 ^f	1336±23 ^d	202±48 ^c	1103±23 ^d	2439±00 ^d
CS55AM(10)+MG(1)	77.5±1.1 ^{ba}	5.9±0.1 ^c	1933±81 ^e	1696±35 ^{cb}	237±47 ^c	2911±30 ^a	4602±05 ^a
CS55AM(20)+MG(0.5)	78.3±0.1 ^{ba}	6.3±0.2 ^{bac}	1392±81 ^{gf}	1268±52 ^d	124±30 ^{dc}	845±79 ^d	2113±131 ^d
CS55AM(20)+MG(1)	76.2±1.7 ^b	6.2±0.0 ^{bac}	1391±66 ^{gf}	1260±52 ^d	131±14 ^{dc}	1070±25 ^d	2330±77 ^d
Coarse grain	79.2±0.0 ^{ba}	6.6±0.2 ^a	1244±32 ^g	1197±33 ^d	047±01 ^c	1065±84 ^d	2261±51 ^d

Legends: Control wheat- durum wheat semolina pasta, CS28AM-28% amylose corn starch, CS55AM-55% amylose corn starch, MG(0.5)- 0.5% mono-glycerides, MG(01)- 1.0% mono-glycerides, PT_c- pasting temperature, PT_m- pasting time, PV- peak viscosity, TV-trough viscosity, BD-breakdown viscosity, SB-setback viscosity, FV-Final viscosity, cP- centipoise, °C-degree Celsius.

Table 2.7: Pasting properties of extruded precooked teff flour pasta samples formulated with corn starch and mono-glycerides.

Treatments	PT _c (°C)	PT _m (mins)	PV (cP)	TV (cP)	BD (cP)	SB (cP)	FV (cP)
Control	50.4±0.5 ^d	6.3±0.1 ^c	1413±103 ^a	1190±101 ^a	224±71 ^a	1482±94 ^a	2672±184 ^a
MG (0.5)	67.0±2.2 ^c	6.6±0.2 ^c	805±45 ^{cb}	744±56 ^{cb}	61±11 ^c	876±6 ^b	1619±49 ^b
CS28AM (10)+MG (0.5)	79.5±0.6 ^a	7.5±1.0 ^{bac}	579±53 ^{ed}	506±74 ^{ced}	73±21 ^c	657±23 ^{cbd}	1163±97 ^{cd}
CS28AM (10)+MG (1)	77.5±1.2 ^a	8.5±0.1 ^a	804±13 ^{cb}	699±16 ^{cb}	106±04 ^{bc}	805±19 ^b	1503±35 ^{cb}
CS28AM (20)+MG (0.5)	77.6±0.1 ^a	6.7±0.2 ^c	776±42 ^{cb}	659±31 ^{cbd}	117±11 ^{bc}	801±02 ^{cb}	1460±29 ^{cb}
CS28AM (20)+MG (1)	78.2±1.1 ^a	8.4±0.2 ^a	913±31 ^b	816±35 ^b	98±04 ^{bc}	886±03 ^b	1702±37 ^b
CS55AM(10)+MG(0.5)	80.0±1.1 ^a	6.3±0.0 ^c	502±113 ^e	443±119 ^{ed}	59±06 ^c	430±66 ^{ed}	873±52 ^{ed}
CS55AM(10)+MG(1)	80.3±1.7 ^a	8.4±0.3 ^a	763±43 ^{cbd}	676±26 ^{cbd}	87±17 ^{bc}	413±121 ^e	1088±147 ^{ed}
CS55AM(20)+MG(0.5)	71.0±1.3 ^{bc}	7.2±0.1 ^{bac}	512±52 ^e	397±52 ^e	116±05 ^{bc}	368±132 ^e	765±179 ^e
CS55AM(20)+MG(1)	75.9±3.5 ^{ba}	8.3±0.1 ^{ba}	621±01 ^{ced}	456±00 ^{ed}	165±01 ^{ba}	393±28 ^e	849±28 ^{ed}
Coarse grain	66.3±1.2 ^d	6.9±0.3 ^{bc}	448±01 ^e	415±02 ^e	034±01 ^c	567±13 ^{ced}	981±16 ^{ed}

Legends: Control wheat- durum wheat semolina pasta, CS28AM-28% amylose corn starch, CS55AM-55% amylose corn starch, MG(0.5)- 0.5% mono-glycerides, MG(01)- 1.0% mono-glycerides, PT_c- pasting temperature, PT_m- pasting time, PV- peak viscosity, TV-trough viscosity, BD-breakdown viscosity, SB-setback viscosity, FV-Final viscosity, cP- centipoise, °C-degree Celsius.

Table 2.8: Pasting properties of raw millet flour blends formulated with corn starch and mono-glycerides.

Treatments	PT _c (°C)	PT _m (mins)	PV (cP)	TV (cP)	BD (cP)	SB (cP)	FV (cP)
Control	63.0±0.6 ^{bac}	5.8±0.1 ^c	2358±23 ^{cbd}	1643±49 ^c	715±47 ^b	1970±153 ^c	3613±194 ^{cb}
MG (0.5)	73.0±0.6 ^{bac}	5.3±0.0 ^{cb}	1637±49 ^{efd}	1049±48 ^c	588±01 ^{cbd}	1279±066 ^{dfe}	2328±114 ^{cd}
CS28AM (10)+MG (0.5)	73.4±0.0 ^{bac}	5.5±0.0 ^{cb}	1742±78 ^{cebd}	1221±58 ^{bac}	521±21 ^{cebd}	1632±037 ^{dce}	2853±95 ^{cbd}
CS28AM (10)+MG (1)	73.1±0.0 ^{bac}	5.7±0.1 ^b	1892±73 ^{cebd}	1477±38 ^{ba}	415±35 ^{ced}	2630±001 ^b	4107±37 ^a
CS28AM (20)+MG (0.5)	73.0±0.6 ^{bac}	5.3±0.1 ^{cb}	2136±15 ^{cb}	1494±79 ^a	642±64 ^{cb}	1206±088 ^{fe}	2702±08 ^{cbd}
CS28AM (20)+MG (1)	74.3±1.1 ^{ba}	5.2±0.0 ^c	2354±44 ^b	1496±21 ^a	859±64 ^b	1310±087 ^{dfe}	2805±66 ^{cbd}
CS55AM(10)+MG(0.5)	75.4±0.7 ^a	5.3± 0.1 ^{cb}	1894±273 ^{cbd}	1300±165 ^{bac}	595±108 ^{cbd}	1439±120 ^{dfce}	2735±284 ^{cbd}
CS55AM(10)+MG(1)	72.0±1.2 ^{bc}	5.5±0.0 ^{cb}	1467±59 ^{efd}	1110±47 ^c	357±12 ^{ced}	1724±202 ^{dc}	2834±249 ^{cbd}
CS55AM(20)+MG(0.5)	72.9±0.1 ^{bac}	5.3±0.1 ^{cb}	1381±24 ^f	1093±25 ^c	288±01 ^{ed}	1114±165 ^f	2207±191 ^d
CS55AM(20)+MG(1)	72.7±0.6 ^{bc}	5.4±0.0 ^{cb}	1416±91 ^{ef}	1194±134 ^{bc}	222±44 ^e	1756±058 ^{dc}	2950±192 ^{cb}
Coarse grain	71.5±0.2 ^c	9.4±0.3 ^a	4584±238 ^a	000±00 ^d	4584±238 ^a	3222±200 ^a	3222±200 ^b

Legends: Control wheat- durum wheat semolina pasta, CS28AM-28% amylose corn starch, CS55AM-55% amylose corn starch, MG (0.5)- 0.5% mono-glycerides, MG (1)- 1.0% mono-glycerides, PT_c- pasting temperature, PT_m- pasting time, PV- peak viscosity, TV- trough viscosity, BD- breakdown viscosity, SB- setback viscosity, FV- Final viscosity, cP- centipoise, °C- degree Celsius.

Table 2.9: Pasting properties of extruded precooked millet flour pasta samples formulated with corn starch and mono-glycerides.

Treatments	PT _c (°C)	PT _m (mins)	PV (cP)	TV (cP)	BD (cP)	SB (cP)	FV (cP)
Control	50.4±0.5 ^d	6.3±0.1 ^{bdc}	1413±103 ^a	1190±101 ^a	224±71 ^a	1482±94 ^{bc}	2672±184 ^a
MG (0.5)	59.5±0.3 ^{cb}	6.8±0.0 ^{bdac}	364±11 ^{cd}	339±06 ^{fde}	25±05 ^b	1014±64 ^{ecd}	1353±70 ^{dc}
CS28AM (10)+MG (0.5)	65.8±0.6 ^a	7.3±0.6 ^{ba}	510±49 ^{cb}	485±35 ^{cd}	24±14 ^b	1257±33 ^{bcd}	1742±68 ^{bc}
CS28AM (10)+MG (1)	61.12.4 ^b	8.0±0.6 ^a	690±30 ^{cb}	486±09 ^{cd}	31±13 ^b	1186±77 ^{bcd}	1671±68 ^{bc}
CS28AM (20)+MG (0.5)	60.3±0.0 ^{cb}	6.1±0.1 ^{bdc}	697±21 ^b	593±08 ^{cb}	97±22 ^b	1499±13 ^{ba}	2092±04 ^{ba}
CS28AM (20)+MG (1)	59.9±0.6 ^{cb}	7.1±0.2 ^{bac}	525±00 ^b	659±23 ^b	38±03 ^b	1694±89 ^a	2353±112 ^a
CS55AM(10)+MG(0.5)	59.5±0.0 ^{cb}	6.8± 0.6 ^{bdac}	523±4 ^{cb}	481±23 ^{cde}	45±23 ^b	1193±80 ^{bcd}	1673±57 ^{bc}
CS55AM(10)+MG(1)	57.0±0.5 ^c	6.7±0.2 ^{bdac}	371±20 ^{cd}	353±37 ^{fde}	19±18 ^b	946±119 ^{ed}	1299±156 ^{dc}
CS55AM(20)+MG(0.5)	68.6±1.1 ^a	5.9±0.4 ^{dc}	248±32 ^d	230±18 ^f	18±13 ^b	395±76 ^f	0625±94 ^e
CS55AM(20)+MG(1)	59.0±0.8 ^{cb}	6.8±0.1 ^{bdac}	402±07 ^{cd}	378±06 ^{fde}	25±1 ^b	981±10 ^{ecd}	1359±16 ^{dc}
Coarse grain	50.0±0.2 ^d	5.6±0.0 ^d	415±08 ^{cd}	332±52 ^{fe}	83±45 ^b	729±18 ^e	1061±35 ^{de}

Legends: Control wheat- durum wheat semolina pasta, CS28AM-28% amylose corn starch, CS55AM-55% amylose corn starch, MG (0.5)- 0.5% mono-glycerides, MG (1)- 1.0% mono-glycerides, PT_c- pasting temperature, PT_m- pasting time, PV- peak viscosity, TV-trough viscosity, BD- breakdown viscosity, SB-setback viscosity, FV-Final viscosity, cP- centipoise, °C-degree Celsius.

Table 2.10: Textural properties of extruded precooked teff and millet pasta formulated with corn starch and mono-glycerides.

Treatments	3PBT (Kg)		Firmness (kg)		Springiness (Kg)	
	Teff	Millet	Teff	Millet	Teff	Millet
Control	4.27±1.43 ^a	6.48±1.71 ^a	10.6±1.1 ^a	9.86±1.1 ^a	1.06±0.0 ^a	1.06±0.0 ^a
MG (0.5)	2.67±0.59 ^b	0.37±0.28 ^c	10.4±1.0 ^a	8.2±1.3 ^{cb}	1.07±0.0 ^a	1.03±0.0 ^a
CS28AM (10)+MG (0.5)	2.84±0.54 ^b	0.11±0.05 ^c	11.1±0.9 ^b	7.9±1.1 ^{cb}	1.05±0.0 ^a	1.01±0.0 ^a
CS28AM (10)+MG (1)	0.94±0.42 ^c	0.21±0.09 ^c	8.5±1.0 ^c	8.7±1.0 ^b	1.04±0.0 ^a	1.00±0.0 ^a
CS28AM (20)+MG (0.5)	2.89±0.92 ^b	1.39±0.89 ^b	9.3±1.5 ^{cd}	5.5±1.1 ^d	1.04±0.0 ^a	1.01±0.0 ^d
CS28AM (20)+MG (1)	1.07±0.47 ^c	0.23±0.13 ^c	8.6±1.3 ^c	6.9±1.3 ^c	1.04±0.0 ^a	1.00±0.0 ^a
CS55AM(10)+MG(0.5)	3.32±0.58 ^b	0.16±0.06 ^c	14.0±0.9 ^e	7.8±1.3 ^{cb}	1.05±0.0 ^a	1.02±0.0 ^a
CS55AM(10)+MG(1)	1.19±0.37 ^c	0.20±0.10 ^c	10.1±1.4 ^a	8.6±1.5 ^b	1.05±0.0 ^a	1.00±0.0 ^a
CS55AM(20)+MG(0.5)	3.27±0.98 ^b	0.33±0.22 ^c	10.4±1.6 ^a	8.1±1.4 ^{cb}	1.05±0.0 ^a	1.01±0.0 ^a
CS55AM(20)+MG(1)	0.91±0.37 ^c	0.12±0.00 ^c	12.9±1.1 ^{be}	8.2±1.0 ^{cb}	1.03±0.0 ^a	1.00±0.0 ^a
Coarse grain	0.92±0.38 ^c	1.96±1.07 ^b	5.4±1.4 ^f	8.9±1.6 ^b	1.01±0.0 ^a	1.00±0.0 ^a
Commercial wheat	1.10±0.41 ^{cb}		5.4±1.5 ^f		1.02±0.0 ^a	
Commercial GF	2.21±0.32 ^{ba}		9.5±1.1 ^b		1.10±0.0 ^a	

Legends: Control wheat- durum wheat semolina pasta, CS28AM-28% amylose corn starch, CS55AM-55% amylose corn starch, MG (0.5)- 0.5% mono-glycerides, MG (1)- 1.0% mono-glycerides, 3PBT- three-point bend test.

Figure 2.1: Scanning electron microscope image of a longitudinal section of teff grain with germ and endosperm.

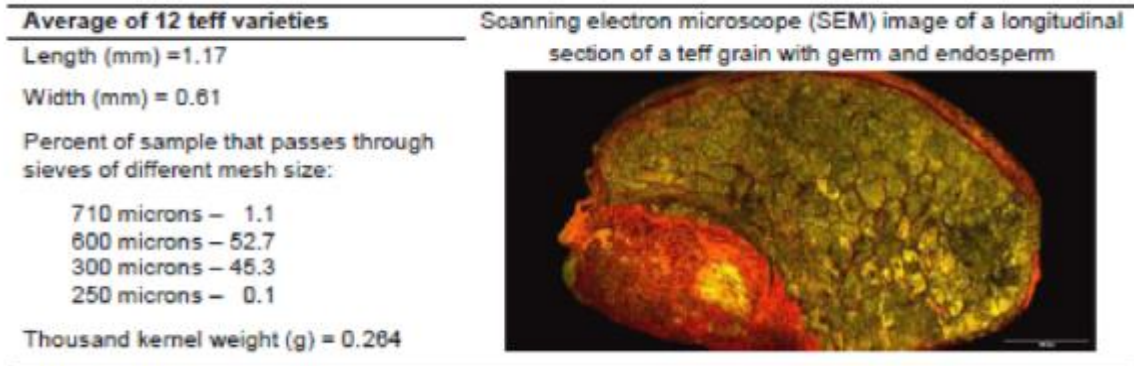
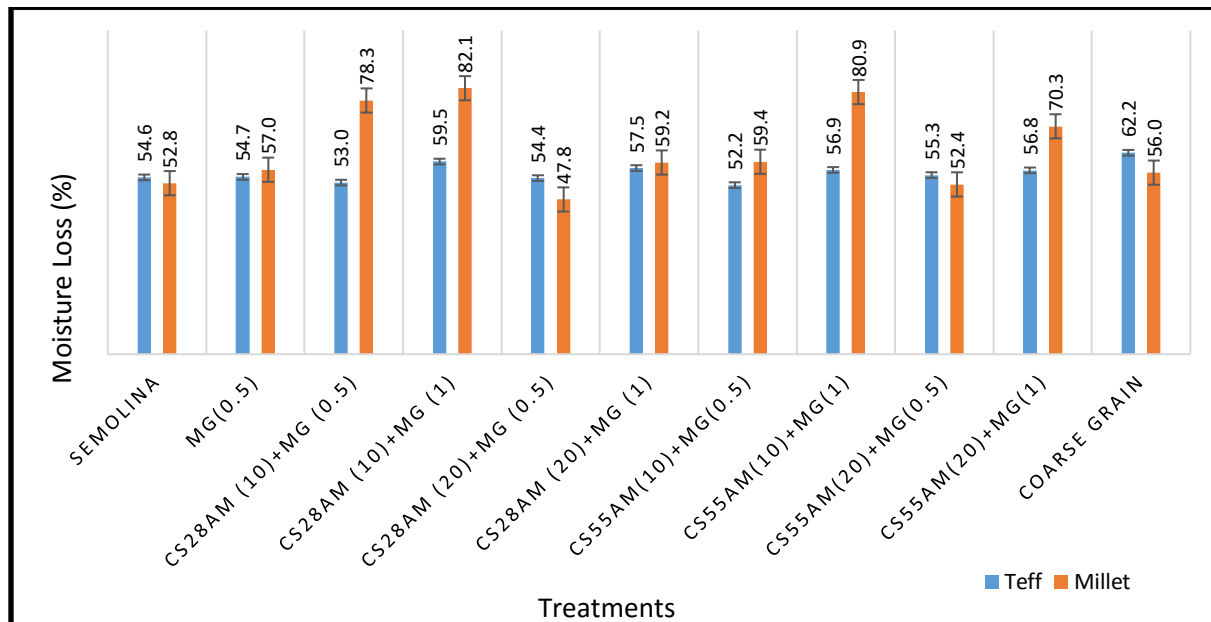


Figure 2.2: Moisture loss of teff and millet pasta during drying at 70°C for 4 hr at high relative humidity.



Legends: Control wheat- durum wheat semolina pasta, CS28AM-28% amylose corn starch, CS55AM-55% amylose corn starch, MG (0.5)- 0.5% mono-glycerides, MG (1)- 1.0% mono-glycerides

Figure 2.3: Average cooking loss of pasta at different levels of mono-glycerides.

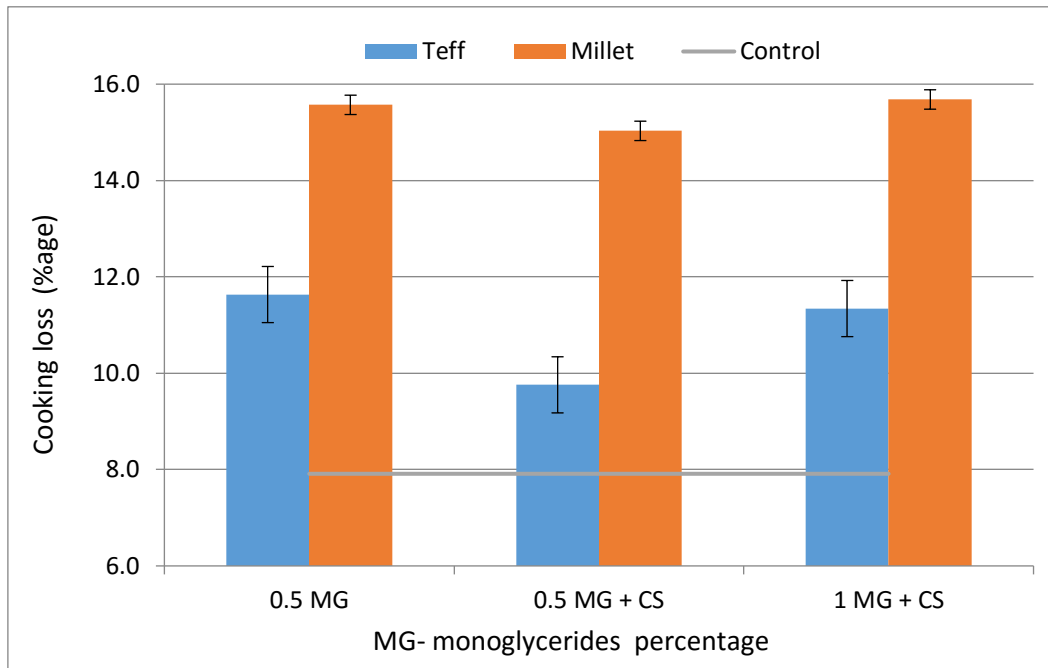


Figure 2.4 Average cooking loss of pasta with native and high amylose corn starch.

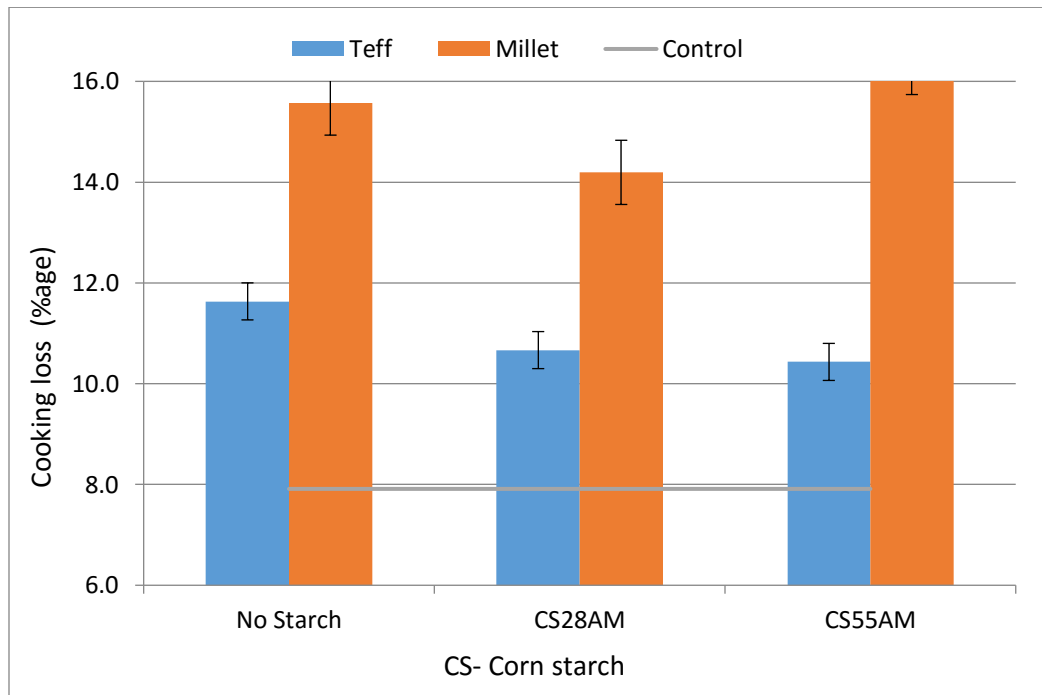


Figure 2.5: Average water absorption of pasta at different levels of mono-glycerides.

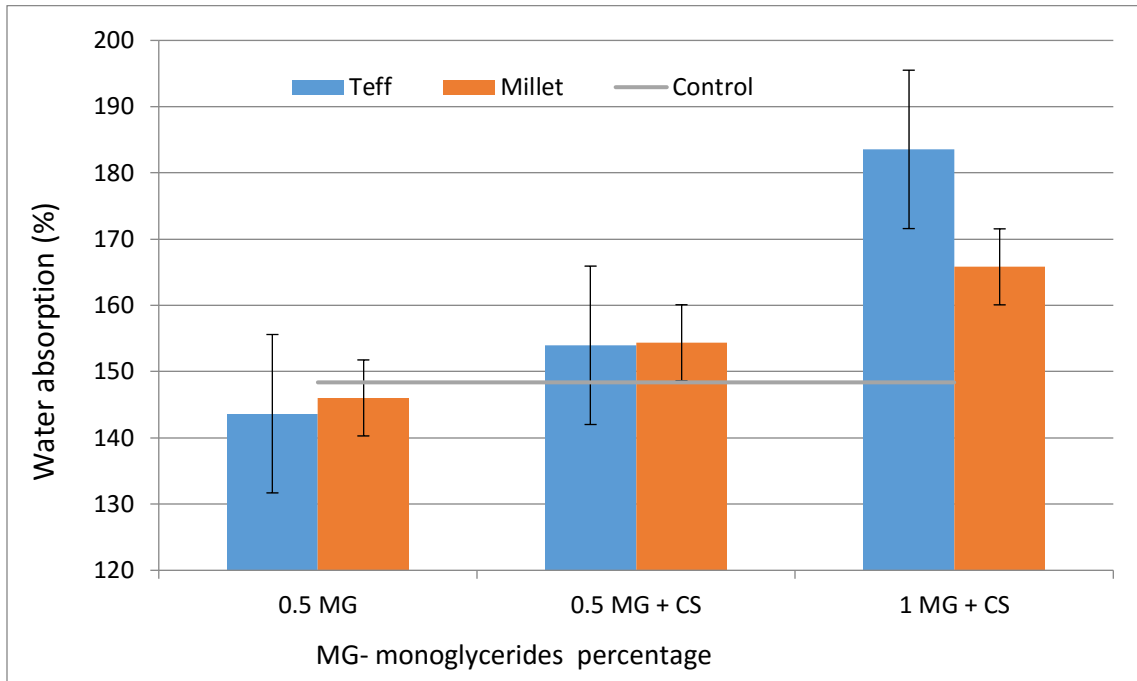


Figure 2.6: Average water absorption of pasta with native and high amylose corn starch.

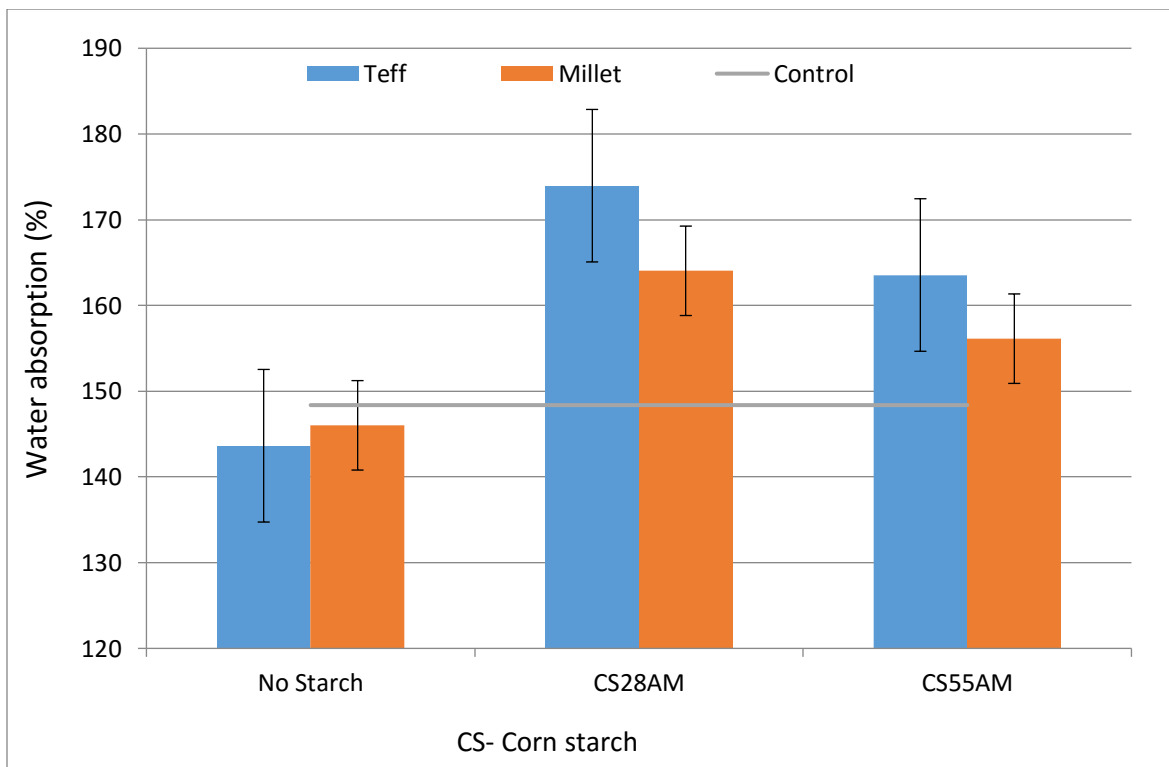
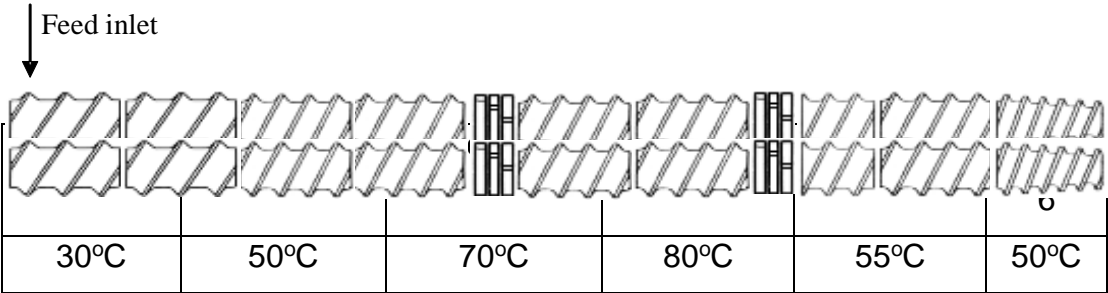


Figure 2.7: Screw profile used in this experiment.



Chapter 3 - Residence time distribution in twin screw extruder at higher moisture and lipid levels in precooked sorghum pasta processing

Abstract

White sorghum flour formulations were prepared by mixing (0.5%, 1% and 1.5%) mono-glycerides (MG) and processed in twin screw extruder at three different in-barrel moistures (40%, 44% and 48%) to measure mean residence time spread using a color trace method. The effect of in-barrel moistures and lipid levels on average residence time distribution (RTD), dispersion numbers and flow pattern was investigated. Both tested conditions were found to have a significant ($p < 0.05$) effect on the mean residence time and other RTD parameters. Increasing the moisture contents significantly reduced the mean residence times and narrowed RTD spread. Increasing lipid levels from 0.5% to 1.5% resulted in higher mean residence times and wider RTD spread. The dispersion number increased with increase in lipid levels, and the flow was turning into mixed flow. No trend clear trend of flow pattern and dispersion number was observed for in-barrel moisture conditions.

Key words: Residence time, extrusion, in-barrel moisture, lipid content, dispersion number, flow patterns

Introduction

Extrusion processing of foods is one of the most efficient continuous cooking, mixing and forming process that has been used increasingly to produce breakfast cereals, baby foods, snacks, pasta, meat analogous, rice, bean and cheese analogues, modified starches etc. Food processing via extrusion inactivates undesirable enzymes that may affect quality and reduce several anti-nutritional factors (Bhandari et al.,2001). Cooking is achieved through the application of heat, either indirectly through jacketed barrels or

directly by steam injection or, and by shearing through mechanical energy (Harper, 1979; Ding et al., 2006). Although, the use of extrusion technology in food industry has increased rapidly but extrusion process is still a complicated multi-input-output system that is yet to be measured. Meuser and Van Lengerich (1984) proposed a simplified system analysis model that sorts extrusion parameters into three groups, namely, process parameters (screw speed, moisture, content, barrel, temperature, screw configuration, die dimensions, raw material characteristics etc.), system parameters (including energy inputs, residence time etc.), and products properties (including color, nutrition, texture, taste etc.). Among these three kinds of parameters, process parameters have effects on the properties of final products by means of affecting extrusion system parameters.

The utilization of sorghum for human food is gaining momentum rapidly. Sorghum crop has qualified as drought tolerance; resistance to mycotoxins and fungi; and survivability in relatively adverse climatic conditions. In several parts of the world sorghum is consumed as a staple crop, and is used as main ingredient to make traditional foods, porridges and alcoholic beverages. In recent developments, the white sorghum grain processed into flour and other products such as snack, cookies and other ethnic foods in several parts of the world. Sorghum protein is non-gluten which offers food choices to gluten allergen consumers. Hence, sorghum has the potential to address both gluten free and sustainability question simultaneously. Pasta is most popular wheat based food that is difficult to develop from non-gluten protein cereals. This research study was also focused on the development of sorghum based gluten free pasta on twin screw extruder.

Pasta is a popular convenience food pan world made from durum wheat or rice using extrusion technology. Suherndro et al. (2002) and Liu et al. (2012) produced noodles from 100 percent sorghum, while Faure (1992) produced pasta with combination of other cereals. Pre-cooked pasta processing is a low shear process performed in twin screw extruders with high in-barrel moisture content (IMC). To produce a high-quality pasta, low cooking loss and good textural characteristics, it is pertinent to optimize the

pasta processing moisture (Fu, 2008; Hou et al., 2010). Similarly, the regulated number of mono-glycerides (MG) also protects the starch granules from degradation which improves the quality attributes of pasta (Charutigon et al., 2008). Both, IMC and MG levels significantly affects the particle process time in extruder.

RTD is a measure of the length of time materials spends in continuous extruder flow system. RTD is one such significant parameter that significantly influences the quality of food products. In general discussion RTD is also referred as process time, every element of feed material is supposed to be exposed to similar residence time, but particles experience variations in residence time due to screw geometry and rheological effects. Several desirable and non-desirable chemical reactions occur during this time which influences product quality by characterizing the reaction time, temperature and shear treatment level of the process (Yu et al., 2014). Meuser et al. (1992) found process variables such as the temperature, screw speed, mass flow rate, screw configuration influence residence time in twin-screw extrusion. To understand the flow pattern inside extruder barrel it is pertinent to understand the distribution of residence time. RTD in extruders also gives information about the degree of mixing, mass flow, velocity profile and life expectancy of fluid inside extruder. (Reitz et al., 2013). RTD studies were used to characterize mixing conditions, flow patterns, and the extent of conversions and reactions of the biopolymers in any plasticizing or cooking extruder (Singh and Rizvi, 1998a). RTD inside extruders is mainly characterized by two parameters; the mean residence time and the exit age distribution of the material inside extruder.

Moisture content not only affects the product properties but also affects the mean residence time, the residence time distribution (RTD), and the flow pattern in extruder. The effect of moisture content on the mean residence time for cereal processing in twin extrusion was studied by (Liang et al., 2012, Kumar et al., 2008 and Sisay et al., 2017). Higher moisture content (19%) resulted in shorter mean residence time and lower RTD spread with reduced gluten based formulation in twin screw extruder (Sisay et al., 2017). Feed moisture had significantly influenced the RTD of particles, and it was negatively correlated with mean residence time and variance. The average mean

residence time was 80 secs at 17% feed moisture content and reduced to 68.9 sec at 19% and further reduced to 63 secs at 21% moisture content. The reason was the conducive environment inside the extruder barrel filled with superheated water and saturated vapor (co-existence) that caused forward push of melt. Chen et al. (2010) found shorter residence time at higher moisture content (28%, 36%, 44%, 52%, and 60%) for soy protein processed in twin screw extruder. Increase in moisture content decreases the viscosity of feed dough in the barrel and lower the energy required to push the melt resulted in lower residence time. Other research studies have shown contrary results at similar moisture levels. Native starch was as feed material processed in twin screw from 16 to 28% moisture content showed increase in mean residence time with increase in moisture content (Kumar et al., 2008). Altomare and Ghossi (1986) found increase in moisture content from 10% to 28.4% raised residence time from 21 to 25 secs in twin screw extruder. Both authors have explained the role of water a plasticizer and viscosity modifier. High feed moisture increased the fluid content of the feed material which caused slippage between screw and barrel resulted in higher residence time.

The addition of lipids causes slippage inside extruder barrel which not only reduces the shear but also increases the mean residence time and RTD spread. An increase in residence time was reported by Phillips and Facone (1988) while processing sorghum meal at 15% and 30% full peanut fat in twin-screw extruder. The reason was increase in lubricity of dough by the higher fat content in formulation. Choudhury and Gautam (2003) found significant increase in mean residence time of rice flour for increase in fish solids concentrations (5%, 10% and 15%). The longer mean residence time was associated with lubricating effect and reduced viscosity of the feed material due to addition of lipids. This study was focused to confirm the effect of both moisture and lipid concentrations individually on residence time in a low shear extrusion process.

Materials and Methods

Raw Materials

Durum wheat semolina was donated by durum processing and milling operation, Tree house, MO. White decorticated coarse sorghum flour with average particle size of 125 μ m of lot number KSU-170427-09 was purchased from Nulife Market, Scott city, Kansas. Distilled mono-glycerides DIMODAN HS K-A was obtained from Danisco USA Inc., New Century, Kansas USA Lot number 1142945586, product no 810773. Iodized salt was purchased from Morton salt Inc, Chicago IL Lot number 23960049. The sorghum flour, salt and mono-glycerides (MG) were blended in appropriate ratios – (98.5% sorghum flour, 1% salt and 0.5% MG), (98% sorghum flour, 1% salt and 1% MG), and (97.5% sorghum flour, 1% salt and 1.5% MG) using a ribbon blender and mixed for 5 minutes. The blends were mixed in batches of 176 pounds. The blends were collected in multi-layered paper bags from the bottom of the mixer by opening a sliding door.

Extrusion Process

The blends of sorghum flour, salt and MG were processed in a pilot scale co-rotating twin screw food extruder X-52 (Wenger Manufacturing Inc., Sabetha, KS, USA) equipped with a differential diameter cylinder preconditioner (DDC2, Wenger Manufacturing Inc., Sabetha, KS, USA). The barrel length 1326 mm is made up five independent zones, fitted with 52 mm diameter screws with an L/D ratio of 25.5:1 (Figure 3.6). The barrel temperatures were increased to 60°C in zone-1, 70 °C in zone-2, 90°C in zone-3, 50°C in zone-4, and 50 in zone-5. The barrel was jacketed and heating was controlled by oil and heating elements. The loss in weight single screw feeder was adjusted to feed 70 kg per hour into the preconditioner. Steam and water were added into the preconditioner, and the amount of Steam added into the preconditioner was ranged between 19.4-21.9 kg/h and water was 5.3-7.7 kg/h. The water flow rate into the preconditioner was controlled by remote flow transmitter, model RFT97121PNUR, RFT9712 S/N 59140 and mass flow sensor model-DS012H205SUP,

sensor S/N 179066, meter type-21, make Micro Motion Inc., CO, USA. The steam flow into the preconditioner was controlled by Digital Pressure High Accuracy Resonate Pressure (DPHARP) sensor, model-EJA110A, style-S1, No-JEJAUR683, make YOKOGAWA. The preconditioner discharge temperature was always maintained above 85°C (maximum was 95°C) with the help of right combination of water and steam in the preconditioner. The preconditioner screw speed was kept constant at 305 RPM.

The IMC moisture content of the feed mix was adjusted to 40%, 44% and 48% (wet basis) based on initial moisture content (10.01g /100g) and additional water injected into the extruder barrel to achieve target moisture levels. The extruder water flow rate in was also controlled by remote flow transmitter model-RFT97121PRU, RFT9712 S/N 70307, SEN S/N 203177 and mass flow sensor model-DS012S100SU, meter type-1, Micro Motion Inc., CO, USA. The steam flow rate into extruder ranged from 7.5 to 9.0 kg/hr, controlled by DPHARP sensor, model-EJA110A, style-S1, No-JEJAUR682, make YOKOGAWA. The screw speed of extruder was kept constant at 252 RPM. Thermocouples mounted at each zone of the extruder barrel were used to record the respective (set and actual) zone temperatures from the control panel. The die temperature and pressure was also recorded directly using thermocouple and pressure gauge respectively. The rotini pasta die, make Maldari, Number- 47637, die head number 55376-1 with 19 (insert) face openings was attached at the exit end of the barrel. The die open area was 0.01796 square inch per insert. A single blade air pressure (75 PSI) rotating face cutter was used to cut the rotini shaped pasta. All the cut pasta pieces were pneumatically conveyed to Wenger Double Pass Dryer/Cooler (Series 4800, Wenger Manufacturing Inc., Sabetha, KS, USA) operating at 71.12°C. The total retention time in the dryer was 59 minutes (8 minutes for top and 40 minutes for bottom belts). Cooling was accomplished by room temperature air with 11 minutes' retention time on cooling belt.

The specific mechanical energy (SME) for each treatment was calculated as follows,

$$SME = \frac{\frac{(\tau - \tau_0)}{100} \times P_{rated} \times \frac{N}{N_{rated}}}{\dot{m}}$$

Where, τ = operating torque (%); τ_0 = no-load torque (%); P_{rated} = rated power (37.3 kW), N = screw speed (rpm); N_{rated} = rated screw speed (336 rpm), and \dot{m} = net mass flow rate of pasta at die exit (kg/s).

Experimental Design

Three different levels of IMC (40% and 44% wet basis) and three levels of MG (0.5%, 1% and 1.5%) were used to observe the influence on residence time distribution. Durum wheat lab produced pasta was used as a control, sorghum based precooked pasta produced in this study was analyzed for cooking loss, water absorption, differential scanning calorimeter (DSC), rapid viscosity analyzer (RVA) and texture profiles analyzer (TPA). The objectives of this study were to determine the effect of processing conditions such as in barrel moisture content and lipid level on the on the RTD and axial mixing behavior (DN) of sorghum flour formulations. The product developed was sorghum based precooked pasta. It is prepared as a value-added product to replace traditional durum wheat pasta with non-traditional sorghum.

Determination of Residence Time

A color tracer was prepared by mixing 5gm of dry food grade green color with 50 gm of feed mix. The whole tracer mix was introduced into the extruder at feed zone (preconditioner downspout) once it reached study state with all processing conditions as indicated by a constant torque, preconditioner steam and water flow, preconditioner discharge temperature, product temperature and die head pressure. Reference samples were collected at the exit die before tracer introduced. The time (t) was started as the tracer introduced into the extruder barrel. Samples were collected as the rotini shaped pasta came out at the die exit each 30 second over a period of 16 minutes (32 samples) trailing the introduction of the tracer into the extruder barrel. Each 30 second sample was collected in a plastic container and placed on an aluminum tray. All the 32 samples were arranged on individual aluminum trays for separation and arranged in series.

Gathered samples were subjected to visual color intensity identification. The collected samples were given scores from 0 to 10 based on the green color concentration, where 10 represented very dark green/ highest level color intensity) and Zero (0) represented no trace of color in the product. The mean value of six reading was considered as a measure of tracer concentration of the green dye.

Fastest particle residence time (FPRT) and extrudate collection time (ECT).

FPRT indicates to the shortest time an extrudate stays in the extruder barrel. It was recorded as the time from the start, when the tracer was introduced into the barrel to the time when the first sign of tracer appeared out of the extruder. The time length between the first appearance of the tracer at the die exit and the time when all the tracer material exited through the extruder is termed as ECT.

Calculating E and F Curves

Experimental measurement of residence time distribution

RTD is determined by a stimulus response technique using a color tracer. Generally, it is determined E(t)-curves, the age distribution of a material in the extruder; and F(t)-curves, the exit age over time of a fluid leaving a vessel (Levenspiel, 1972). Stimulus response of trace is usually used to plot the RTD curves. A pulse tracer (color dye) was introduced into the extruder barrel from feeder at time t=0. The dye concentration in the extrudates (C_i) is measured by manual observation. Lee (2012), calculated C_i from color values, and used dye concentrations values to plot standard curves. The tracer concentration is required to be normalized at each point (in time) by dividing them by the summation of tracer concentration passing through the system. The E(t) curve was obtained by the following equation (Levenspiel, 1999):

$$E(t) = \frac{C(t)}{\int_0^{\infty} C(t)dt} \cong \frac{C_i}{\sum_{i=0}^{\infty} C_i \Delta t_i}$$

(1)

C is the tracer (color) concentration at time t.

E(t) values were obtained by output concentration C divide by the total concentration C₀. C_t is the concentration of green color at residence time t, the summation of all C's collected over the extrudates collection time represented the total C. And, the fraction of tracer concentration E(t) at various residence time t, is measured by utilizing E-curves.

The F(t) curve represents the cumulative particle concentration distribution function of the exit stream at any time (t). It is obtained as

$$F(t) = \int_0^t E(t)dt \cong \frac{\sum_0^t C(t) \Delta t_i}{\sum_0^\infty C(t) \Delta t_i} \quad (2)$$

The mean residence time (t_m) is calculated by following equation (Levenspiel, 1999):

$$t_m = \int_0^\infty E(t)dt \cong \frac{\int_0^\infty C(t) t dt}{\int_0^\infty C(t) dt} = \frac{\sum_{i=0}^\infty t_i C_i \Delta t}{\sum_{i=0}^\infty C_i \Delta t} \quad (3)$$

The variance (σ²) is calculated by squaring the spread of distribution (Levenspiel, 1999):

$$\sigma^2 = \sum_{i=0}^\infty (t_i - t_m)^2 E(t_i) \quad (4)$$

To compare the flow patterns at different processing conditions, the individual residence time was divided by mean residence time to normalized all values of particle residence time (Kumar et al. 2008). The normalized time, E-curve and F-curves are obtained as follows (Levenspiel, 1999):

$$\text{As, } \theta = \frac{t}{t_m}$$

E-curve was normalized as follows

$$E(\theta) = \frac{C_\theta}{C_0} = \frac{C_\theta}{\sum_{\theta=0}^{\infty} C_\theta \Delta t} = t_m \times E(t) \quad (5)$$

F-curve was normalized as follows

$$F(\theta) = \int_0^\theta E(\theta) d\theta = \frac{\sum_0^\theta C(\theta) d(\theta)}{C_0} = \frac{\sum_{i=0}^\theta C_i \Delta\theta}{\sum_{i=0}^{\infty} C_i \Delta\theta} = F(t) \quad (6)$$

And variance is normalized as follows

$$\sigma^2 = \frac{\sigma^2}{t_m^2} \quad (7)$$

Most of the mixing in the extruder is achieved by laminar mixing actions, because it is difficult to quantify the axial mixing directly from RTD curves. Kumar et al. (2006) found out the dispersion number (D_N) is a good index for the measurement of axial mixing in an extruder. The dispersion number (D_N) is the reciprocal of Peclet number used to measure the extent of axial dispersion (Kumar et al., 2006).

The dispersion number (D_N) defined as D/uL ,

where D is the diffusivity, u is the flow rate and L is the length of the vessel.

The flow is defined as plug flow when D_N approaches zero, the dispersion is negligible. The flow is mixed flow when dispersion number (D_N) approaches infinity, the dispersion number is significant to consider. Extrusion is closed vessel, and for closed vessel, the dispersion number (D_N) can be calculated using mean residence time and variance values of the curve.

Equation to calculate dispersion using mean residence time and variance values as follows:

$$\sigma_{\theta}^2 = \frac{\sigma^2}{t_m^2} = 2 \frac{D}{uL} - 2 \left(\frac{D}{uL} \right)^2 (1 - e^{-\frac{uL}{D}})$$

(8)

The dispersion number for each treatment was calculated by error and trial method. The RTD and mixing behavior of low shear precooked sorghum pasta has not been studied earlier. The objective of this research work was to optimize the sorghum pasta processing conditions such as in barrel moisture content and lipids (mono-glycerides) levels and its effect on the residence time distribution and mixing behavior (D_N) in twin screw extruder.

Statistical Analysis

All the results were analyzed using analysis of variance (ANOVA) with general linear model procedure (SAS version 9.1, SAS Institute, Cary, North Carolina, USA). When significant effects ($p < 0.05$) were indicated by ANOVA, Tukey pairwise comparisons were conducted to distinguish which treatments differed significantly ($p < 0.05$). Pearson Correlations was used to establish correlation values.

Results and Discussion

E and F-curves

Influence of moisture content

Figure 3.3, shows the influence of different process moisture contents on E and F curves. The Figure 3.3a and b represent the E-distribution and time (t), while Figure 3.3c and d represents cumulative normalized trace concentration $F(\theta)$ with normalized time $t(\theta)$. The treatment with 48% moisture content had the fastest particle residence time (FPRT) and the shortest spread distribution. Moisture content was found not to be significant ($p > 0.05$) for FPRT and ECT (Table 3.1a and b). Liang et al. (2012) reported

no significant difference in ECT, FPRT and mean residence time variance at 25%, 30% and 35% moisture content in twin extruder. High moisture content (48%) has decreased the ECT to spread and shifted the curves towards left, whereas low moisture (40%) content has increased the spread distribution. Low moisture resulted in lower E(t) peaks, while high moisture resulted higher peaks. As mentioned earlier, at higher process moisture there is a less competition for water absorption between starch, protein and fiber. The surplus water facilitates lubricating effect inside extruder. Similar trends of E(t) at higher moisture content were observed by Omeire et al. (2013) and Yu et al. (2014). While the qualitative nature is similar the peaks E(t) increase with increase in moisture content but decreased the spread. Similar, decreasing trends of ECT with increase in moisture content were observed by Liang et al. (2012).

The normalized F-curves for different moisture levels are represented in Figure 3.3c and d. Increase in moisture content reduced the mean residence time and shifted E and F curves on the left. The 48% moisture treatment had the shortest time to reach 100% accumulation, while 40% moisture treatment spent the longest time to go out of the extruder. Increasing the moisture content resulted in a more rapid accumulation of the particles and a higher peak of the normalized output concentration, and in shorter residence time of the sample in the extruder (Yu et al., 2014).

Influence of lipids content

The effect of different lipids levels on E and F curves are shown in Figure 3.4. E-curves are represented in Figure 3.4a and b and normalized F-curve is represented in figure 3.4c and d. The mean ECT values were not significantly different ($p>0.05$) for different lipid levels. The treatment with highest lipid content had the largest spread and lowest lipid content had the shortest spread. The increase in lipid content from 0.5% to 1.5% has increased the ECT spread. However, lipid content has inversely effected the color tracer peaks E(t), low lipid content resulted in high peak values and vice-versa. The peak of E(t) curves decreased from 0.179 to 0.111 and shifted towards right with increasing amount of lipids content in blend. The results agree with the findings of Choudhury and Gautam (2003).

F(θ) curves clearly differentiate the cumulative particle concentration distribution for different levels of lipids. Treatment formulated with 1.5% MG took longest time to reach 100% accumulation, while lowest lipid content (0.5%MG) took shortest time to exit extruder. The increase in lipid content resulted in a slow accumulation of particles and leads to higher mean residence time. The SME inputs decreased upon increasing lipids content leads to result in viscosity. The addition of lipid caused a lubricating effect thereby causing more slippage rather than positive conveyance of the material. Choudhury and Gautam (2003) found delayed in particle accumulation and longer time to reach complete accumulation by increasing levels of fat.

Residence Time and Variance at Different In-barrel Moistures

The mean residence time and variance values of each treatment are shown in Table 3.1a and b. The mean residence time was significantly affected by increasing the moisture content from 40% to 48% ($p < 0.05$). The mean residence time was 4.78 mins at 40% IMC; 3.59 mins at 44% and 3.87 mins at 48% IMC (Table 3.1a). The results clearly shown a trend of decrease in mean residence time as moisture levels increased. Similar trend of decrease in residence time by increasing the moisture content was observed in repetition. The mean residence time was 4.47 mins at 40% IMC; 3.89 mins at 44% and 3.74 mins at 48% IMC (Table 3.1b). The values of mean residence time were significantly different ($p < 0.05$) for each moisture level (Table 3.1a and b). Yu et al. (2012) found significant increase in mean residence time from 62 to 87 secs on decreasing the moisture content from 35% to 25%. A significant negative correlation ($R^2 = 0.54$) for first (Figure 3.5a) and second ($R^2 = 0.90$) experiment (Figure 3.5b) was found between moisture content and mean residence time in both experiments.

Residence time of feed material inside the extruder is supposed to be a degree indicator of raw material experiencing shearing, heating, shaping, mixing and reaction. It was found that increasing moisture content could result in accelerating the flow speed of melt coming out from extruder. The possible reasoning could be the increase in moisture content reduces the viscosity of the melt inside the barrel. In other words, the

melt is more fluid at higher process moisture content (Flecher et al., 1985). Since we have added water directly into the heated barrel it does not provide enough time to feed material to absorb added water completely. The extruder barrel is a closed vessel; hence it is impossible for the heated water to evaporate. The heated water gets mixed with melt, and reduces the restriction of flow which results in lower mean residence time. When moisture content is low, majority of it get utilized during protein denaturation and starch gelatinization, means less free water is available for melt fluid. Hence the particle (lump) formation and forward push motion was suppressed which resulted into increase in residence time.

Seker (2005) explained the effect of moisture content on mean residence time, which is usually considered in two opposite ways. First, increasing the moisture content of feed material results in the decrease of viscosity of feed material in the barrel of an extruder, and reduces the torque required to pump the melt through the die. The short time cooking process doesn't allow the starch granules to swell which further lowers the specific energy required to drive the melt through the die (Kirby et al., 1988). Similar, trend of drop in SME with increase in moisture content was observed (Table 3.1a and b). Second, temperature in the die due to viscous dissipation is lower, and the lower temperature of feed increases the viscosity at the die, which tends to increase the restriction of flow through the die. The effect of moisture content on the mean residence time is expected to be the result of these two-opposite effects of moisture content on rheology of feed material in the barrel and die of the extruder. Chen et al. (2010) found significant decrease in dough viscosity at the die with moisture content increasing. Therefore, increasing the moisture content would result in lowering the residence of melt inside extruder.

High moisture content resulted in lower viscosity of feed material and the relationship between feed moisture content and average apparent viscosity was found to be exponential (Lo et al., 1998). This is consistent with previous results (Bhattacharya and Hanna, 1987; Senouci and Smith, 1988). Increasing feed moisture concentration resulted in a decrease in the SME. This was related to the reduction of both the mean residence time from 51.8secs at 17% moisture to 47.8 secs at 19.6% moisture (Lo,

1996) and the apparent viscosity of the melted cornmeal in the extruder. These results are confirmed by previous studies (Chang and Halek, 1991; Lu, 1992). The non-evaporation of superheated water and saturated water vapor facilitated forward push of particle inside barrel which decreased the mean residence time by 17 secs with 4% increase in moisture content (Sisay et al., 2017). Gogoi and Yam (1994) found out significant reduction in mean residence time by increasing moisture content but specific reason was mentioned.

However, in some cases, we also observed that residence time could increase with moisture content quite significantly. Altomare and Ghossi (1986) reported that water acts a plasticizer in extrusion processing; it promotes lubrication between screw and barrel. Increasing the moisture content from 10.0% to 28.4% lowered the viscosity of feed material resulted in longer mean residence time. Lin and Armstrong (1990) showed that decreasing the feed moisture from 30% to 20% in a Brabender DSE35 twin-screw extruder increased viscosity and therefore, decreased the mean residence time from 45.3 secs to 31.7 secs. Similar results were showed by Kumar et al., 2008; when native starch was processed in twin screw extruder, caused increase in mean residence time by increase in moisture percentages from 16% to 28%. Some studies have been inconclusive on the effect of moisture content on mean residence time. Such as, the mean residence time was not significantly affected with increase the moisture content from 28.5% to 41.2% (Seker, 2005). Gogoi and Yam (1994) reported that moisture content had only marginal effect on the residence time. These results contrast from our study, which may be due to the use of different moisture contents, raw materials, and starch to protein ratio, process conditions, and different design of the extruder system. The other possible reason could be the design of extrusion system. Single screw extruders are not designed to handle various process conditions such as high moisture levels. It may be possible that increase in feed moisture content could cause slippage in single screw extruder whereas twin screws can easily convey the material due to its intermeshing scraping effect. Hence, twin screw could perhaps nullify the slippage effect of high moisture concentration.

The mean residence time variance values were not significantly different ($p > 0.05$) for

three moisture levels (Table 3.1a and b). A trend of increase in residence time variance with increase in mean residence time and vice versa was observed at different in-barrel moisture conditions. This could be due to the change in mixing behavior. The mixing pattern might be moving towards plug flow from mixed flow at high in-barrel moisture which could have led to high variance at high mean residence time and vice-versa. Hence, higher in-barrel moisture resulted into low mean residence time, low variance and plug flow. This is in agreement with Sisay et al. (2017) and van van Zuilichem et al. (1988), they reported the flow approached plug flow at higher feed moisture contents. The mean variance was 626 at 40% moisture, 169 at 44% moisture content and 210 at 48% moisture content (Table 3.1a). The average mean variance values decrease by 66.5% when moisture content was raised from 40% to 48%. The mean values of variance are 321 at 40% moisture content, 465 at 44% at moisture content and 260 at 48% moisture content (Table 3.1b). The mean variance decreased by 23.6% when moisture content was increased from 40% to 48%. Similar trends of drop in mean variance (550, 412 and 342) at higher moisture content (17%, 19% and 21%) were also found by (Sisay et al., 2017). The reason explained by Sisay et al. (2017) was that an increase in feed moisture content decreases the SME required to push wet mass through the die, and results in reducing the friction between the raw material and screw shaft and extruder barrel. Similar trends of decrease in variance at higher moisture levels (25%, 30%, and 35%) were reported Yu et al. (2012).

Residence Time and Variance at Different Lipid Levels

The average values of mean residence time were 3.87 mins at 0.5% MG, 4.48 mins at 1% MG and 4.70 mins 1.5% MG (Table 3.2a). A significant positive correlation ($R^2=0.93$) during first (Figure 3.5c) and ($R^2=0.94$) in second experiment (Figure 3.5d) was found between mean residence time and lipid percentage. The average values of mean residence time were 3.74 mins at 0.5% MG, 3.98 mins at 1% MG and 4.58 mins at 1.5% MG in second experiment (Table 3.2b). The average values of residence time were not significantly different ($p>0.05$). A trend of increase in mean residence time of particles with increase in lipid content was observed in both the experiments. The

addition of lipids produced a lubricating effect and increases the slippage of the melt. The lubricating effect that cause slippage rather than positive conveyance of melt, lowered the SME input, and resulted in longer residence time (Kirby et al., 1988).

A trend of lowered SME inputs and higher mean residence time were represented due to higher percentages of lipids (Table 3.2a and b). Residence time is inversely related to flow rate in the extruder, the latter being a function of extruder and die dimensions-geometry; directly proportional to dough viscosity in the metering flights and inversely proportional to viscosity in the die for Newtonian flow (Harper, 1981). Since flow rate, screw RPM and screw-die geometry were constant through this experiment, residence time should have been purely a function of dough viscosity and slippage inside the barrel. Phillips and Falcone (1988) found longer residence time at higher fat content of feed composition due to lubrication of melt. Similar results of longer residence time at higher levels of lipids were reported by Choudhury and Gautam (2003), Altomare and Ghossi (1986) and Lin and Armstrong (1990).

The mean residence time variance values were 210 at 0.5% MG, increased by 259% - 753 at 1%, and increased by 19% -896 at 1.5% MG (Table 3.2a). The mean residence time variance values were not significantly different (p -value>0.05) for three lipid levels (Table 3.2a and b). A trend of increase in residence time variance with increase in mean residence time at higher lipid concentration was observed. The possible reason could be the change in mixing behavior. Dispersion number increased as the lipids content was increased and the flow patterns was turning into mixed flow. Therefore, higher lipid levels resulted into high mean residence time, high variance and mixed flow. The average residence variance values were 260 at 0.5% MG, 371 at 1% MG, and 657 at 1.5% MG in repetition (Table 3.2b). The mean variance values increased by 43% when lipid content was increased from 0.5% to 1% and 77% increase from 1% to 1.5% rise in lipid levels. The possible reason for increase in variance with increase lipid levels could be drop in barrel pressure caused a lubricating effect thereby causing more slippage rather than positive conveyance of the material. The slippage effect may have resulted in discontinuous flow of colored material at the die. Choudhary and Gautam (2003) also reported increasing trends of variance with increasing percentage of lipid.

Dispersion Number and Flow Pattern

The dispersion numbers (D_N) are shown in Table 3.3. The dispersion numbers ranged from 553 to 9094 for first experiment. The lowest D_N of 553 at 44% moisture content and highest was 4294 at 40% moisture content. During the second experiment the lowest D_N of 1205 was at 48% moisture content and highest was 3571 at 44% moisture content. There was no clear trend of D_N with increase or decrease in process moisture concentrations.

The lowest D_N for different lipids was 734 at 0.5% MG and highest 9094 at 1.5% MG for first experiment; and lowest 1205 at 0.5% MG and highest 5141 at 1.5% MG for second experiment. A trend of increase in D_N with increase in lipid content was observed during both experiments. The flow was moving toward a mixed flow with an increase of lipid concentration and toward plug flow with a decrease of lipid concentration.

Conclusion

The mean residence time and its variance of sorghum flour mix formulated salt and mono-glycerides was influenced significantly by increase in process moisture content from 40% to 48% and lipid content from 0.5% to 1.5% in the twin screw extruder. Increasing moisture content reduced the mean residence time and its variance. While by increasing lipid content in the formulation increased the mean residence time. The RTD spread was wider at low moisture and higher lipid levels.

Increasing in the moisture content decreased the ECT spread and resulted in higher tracer concentration peaks. The ECT and $F(\theta)$ curves were shifted towards right at higher moistures, the 48% moisture treatment took shortest time to reach 100% accumulation to exit the die. Increase in lipid content of treatments slower the accumulation of particles and resulted in higher mean residence time. The treatment with 1.5% MG content took the highest time to reach 100% accumulation whereas 0.5%

MG content took shortest time to exit the extruder. The residence time results obtained in this study can be used for modeling; scale-up of extrusion process; defining proper cooking and microbial inactivation time for low shear processed like pasta.

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Table 3.1: Operating conditions at different moisture contents.

a): Operating conditions for first experiment and results at different moisture contents.

Run	IMC	MG	t_m	σ^2	SME	FPRT	ECT
1	40	0.5	4.78 ^a	626 ^a	162	105 ^a	810 ^a
2	44	0.5	3.59 ^b	169 ^a	91	105 ^a	540 ^a
3	48	0.5	3.87 ^{ab}	210 ^a	56	105 ^a	540 ^a

IMC- In-barrel moisture content (%), MG- mono glycerides (MG), t_m – mean residence time (mins), σ^2 - variance, SME- specific mechanical energy (kj/kg), FPRT-Fastest particle residence time and ECT- extrudate collection time.

b): Operating conditions for second experiment and results at different moisture contents.

Run	IMC	MG	t_m	σ^2	SME	FPRT	ECT
1	40	0.5	4.47 ^a	321 ^a	176	135 ^a	600 ^a
2	44	0.5	3.89 ^b	465 ^a	130	105 ^a	690 ^a
3	48	0.5	3.74 ^{ab}	260 ^a	91	105 ^a	510 ^a

IMC- In-barrel moisture content (%), MG- mono glycerides (MG), t_m – mean residence time (mins), σ^2 - variance, SME- specific mechanical energy (kj/kg), FPRT-Fastest particle residence time and ECT- extrudate collection time.

Table 3.2: Operating conditions at different lipid contents

a): Operating conditions for first experiment and results at different lipid contents.

Run	IMC	MG	t_m	σ^2	SME	FPRT	ECT
1	48	0.5	3.87 ^b	210 ^a	56	105 ^a	540 ^a
2	48	1.0	4.48 ^{ab}	753 ^a	58	75 ^a	870 ^a
3	48	1.5	4.70 ^a	896 ^a	51	75 ^a	870 ^a

IMC- In-barrel moisture content (%), MG- mono glycerides (MG), t_m – mean residence time (mins), σ^2 - variance, SME- specific mechanical energy (kj/kg), FPRT-Fastest particle residence time and ECT- extrudate collection time.

b): Operating conditions for second experiment and results at different lipid contents.

Run	IMC	MG	t_m	σ^2	SME	FPRT	ECT
1	48	0.5	3.74 ^b	260 ^a	91	105 ^a	510 ^a
2	48	1.0	3.98 ^{ab}	371 ^a	71	105 ^a	630 ^a
3	48	1.5	4.58 ^a	657 ^a	65	105 ^a	840 ^a

IMC- In-barrel moisture content (%), MG- mono glycerides (MG), t_m – mean residence time (mins), σ^2 - variance, SME- specific mechanical energy (kj/kg), FPRT-Fastest particle residence time and ECT- extrudate collection time.

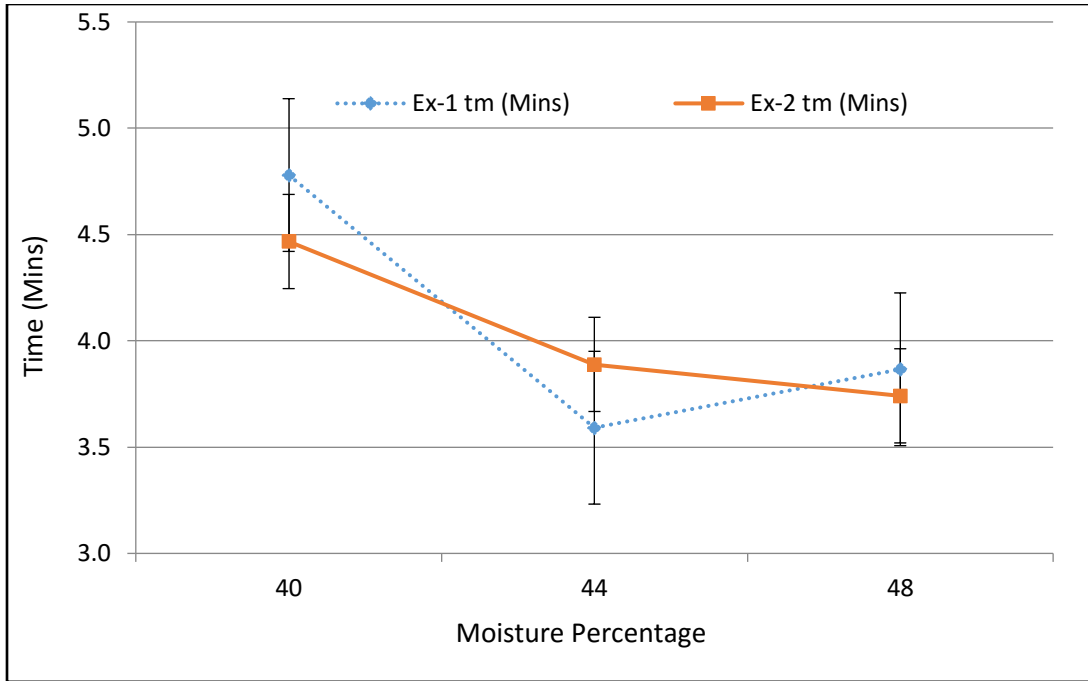
Table 3.3:Dispersion numbers for both experiments.

Run	IMC	MG	D _{N1}	D _{N2}
1	40	0.5	4294	1292
2	44	0.5	553	3571
3	48	0.5	734	1205
4	48	1.0	7055	2169
5	48	1.5	9094	5141

IMC- In-barrel moisture content (%), MG- mono glycerides (MG), D_{N1}- dispersion numbers for first experiment, D_{N2}- dispersion number for second experiment.

Figure 3.1: Effect of different conditions of mean residence time.

a) Effect of in-barrel moisture content on mean residence time.



b) Effect of in-barrel lipid content on mean residence time.

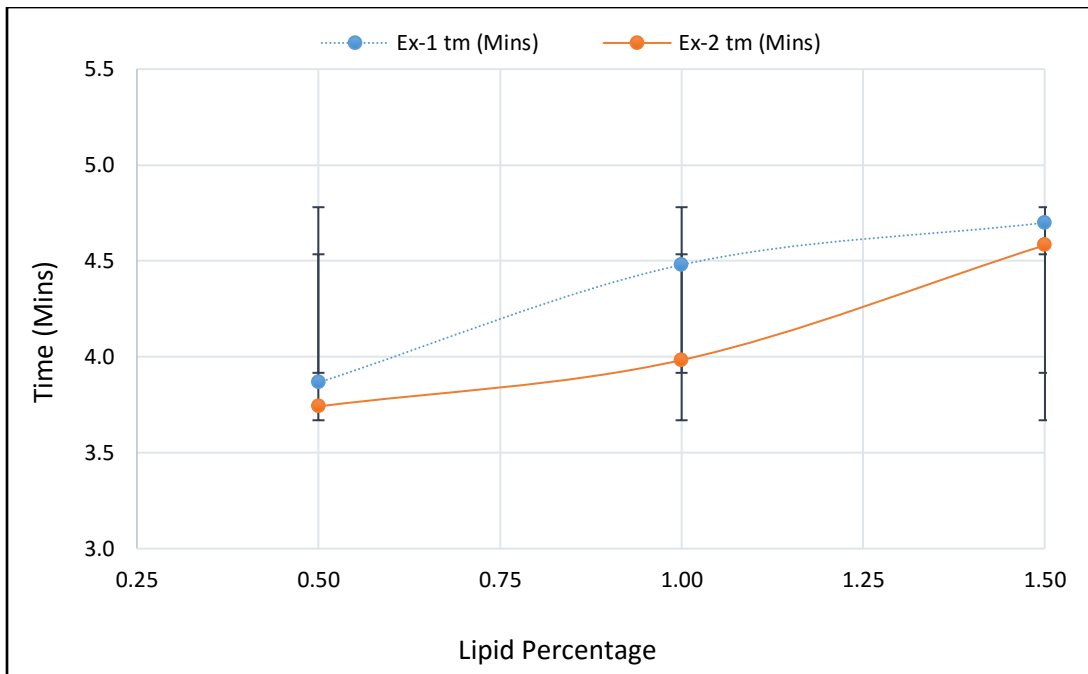
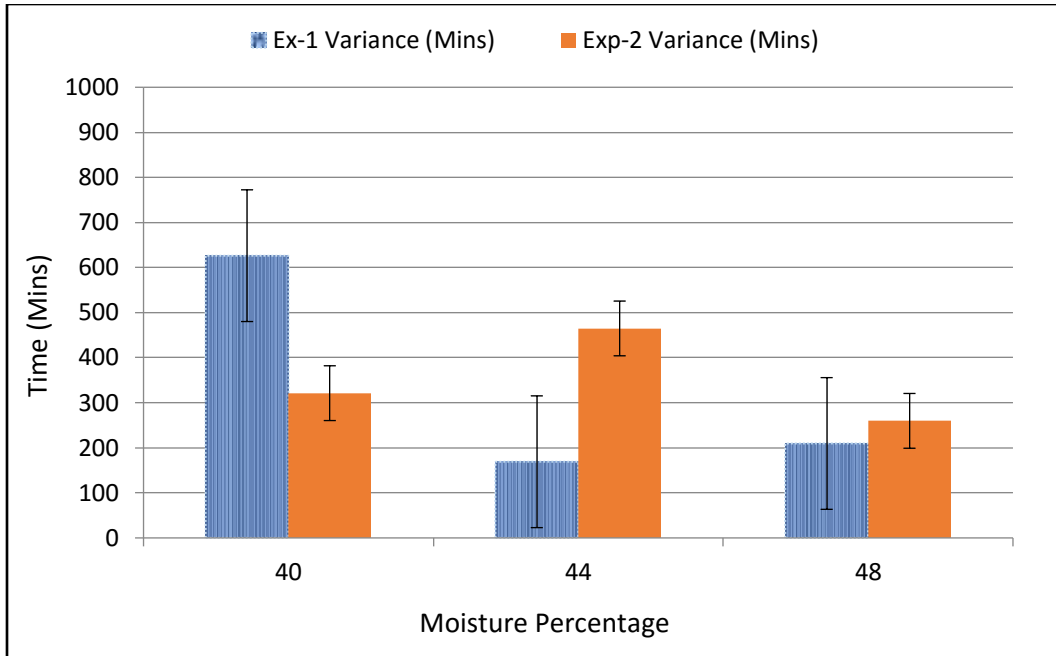


Figure 3.2: Effect of different conditions of mean variance.

a) Effect of in-barrel moisture content on mean variance time.



b) Effect of in-barrel lipid content on variance time.

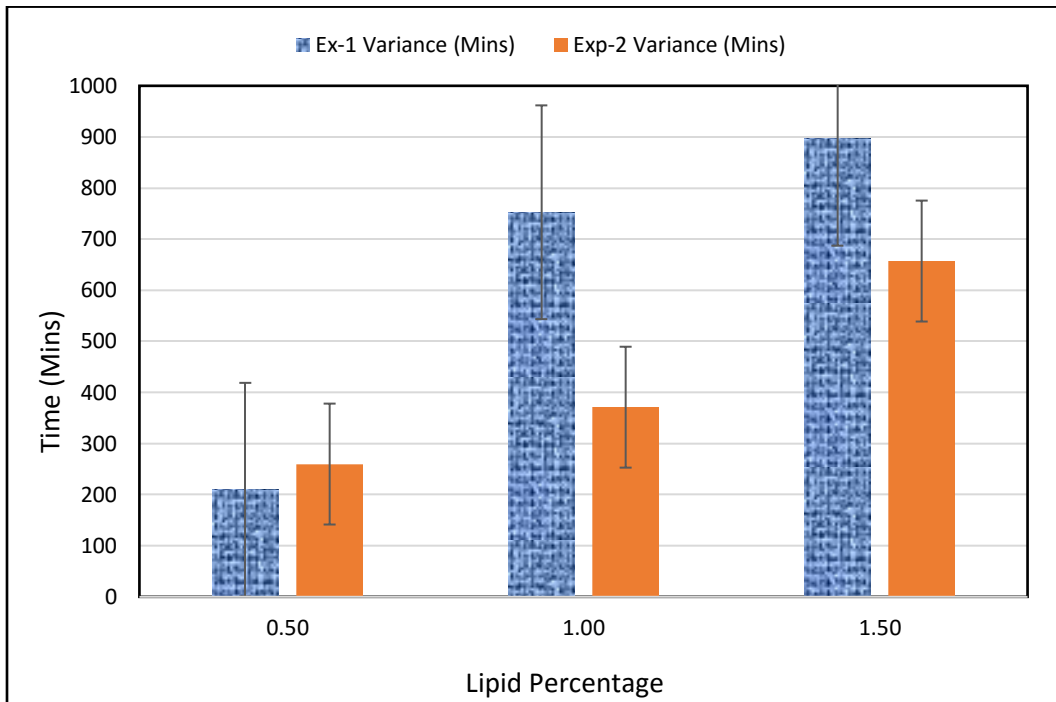
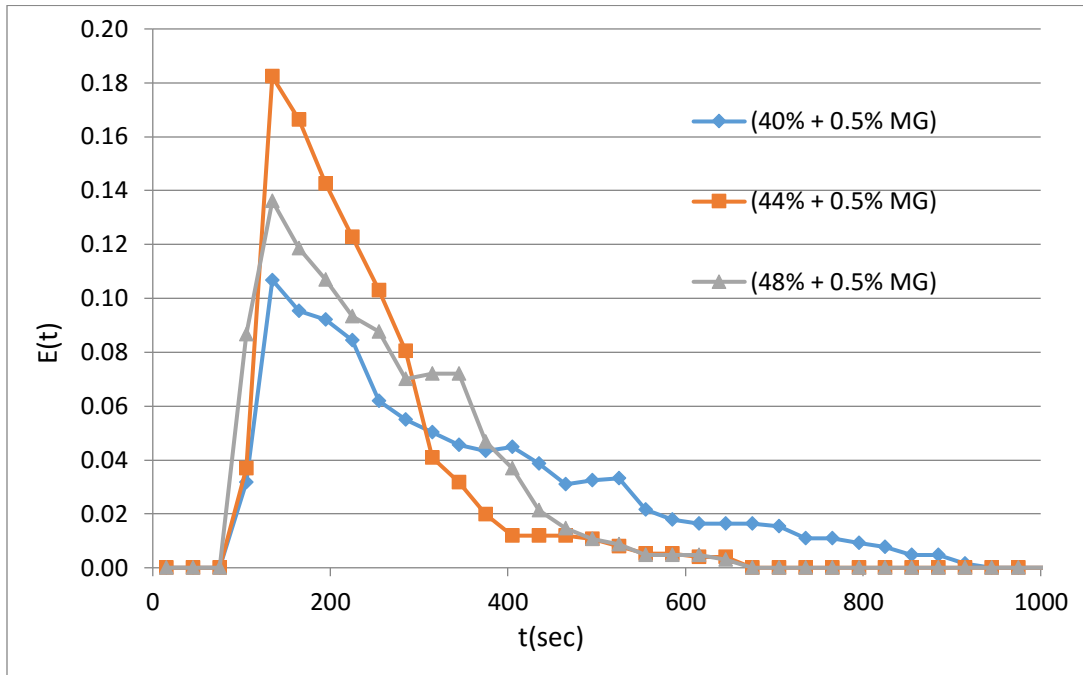
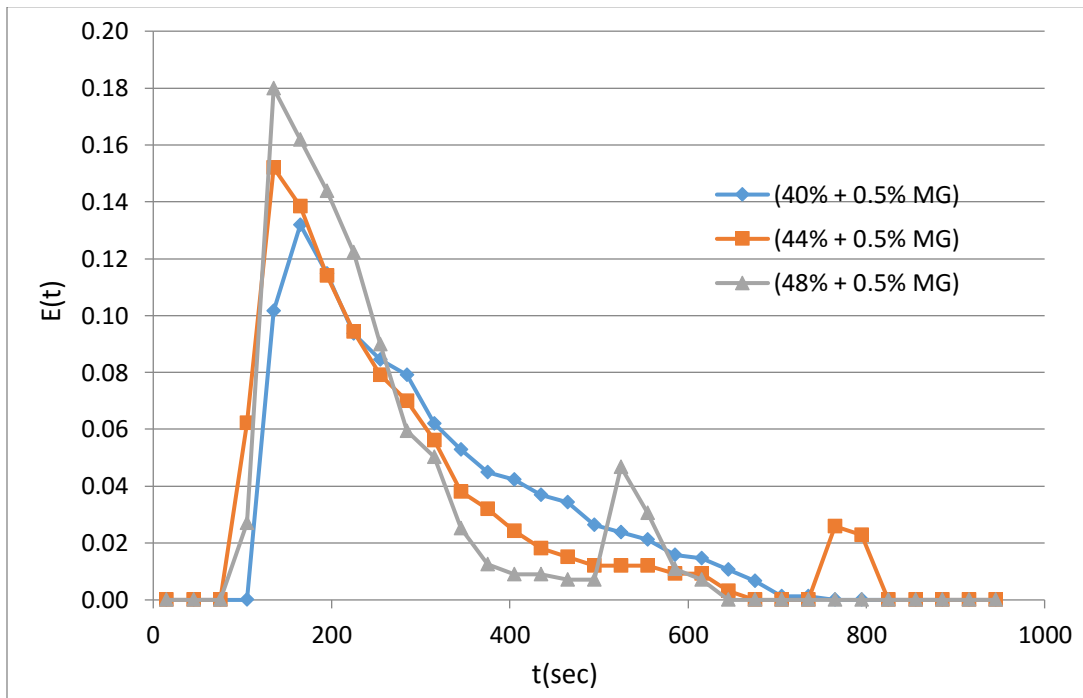


Figure 3.3: Effect of different in barrel conditions on E(t) and F(θ) curves.

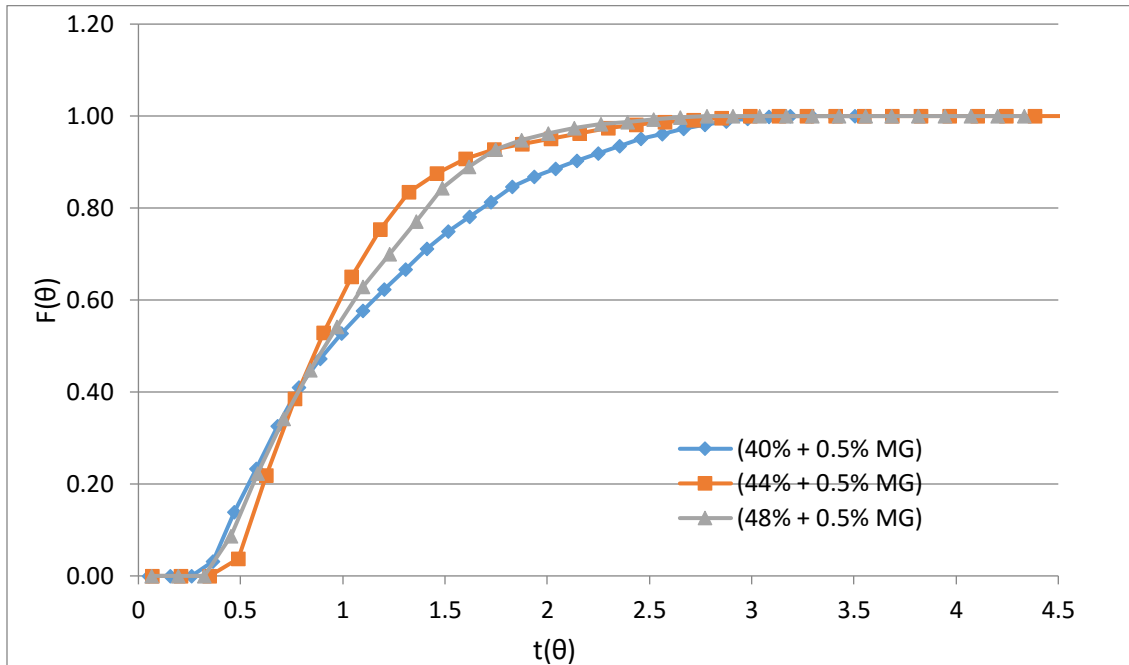
a) Effect of in-barrel moisture content on E(t) curves distribution with time for first experiment.



b) Effect of in-barrel moisture content on E(t) curves distribution with time for second experiment.



c) Effect of in-barrel moisture content on $F(\theta)$ curves distribution with normalized time for first experiment.



d) Effect of in-barrel moisture content on $F(\theta)$ curves distribution with normalized time for the second experiment.

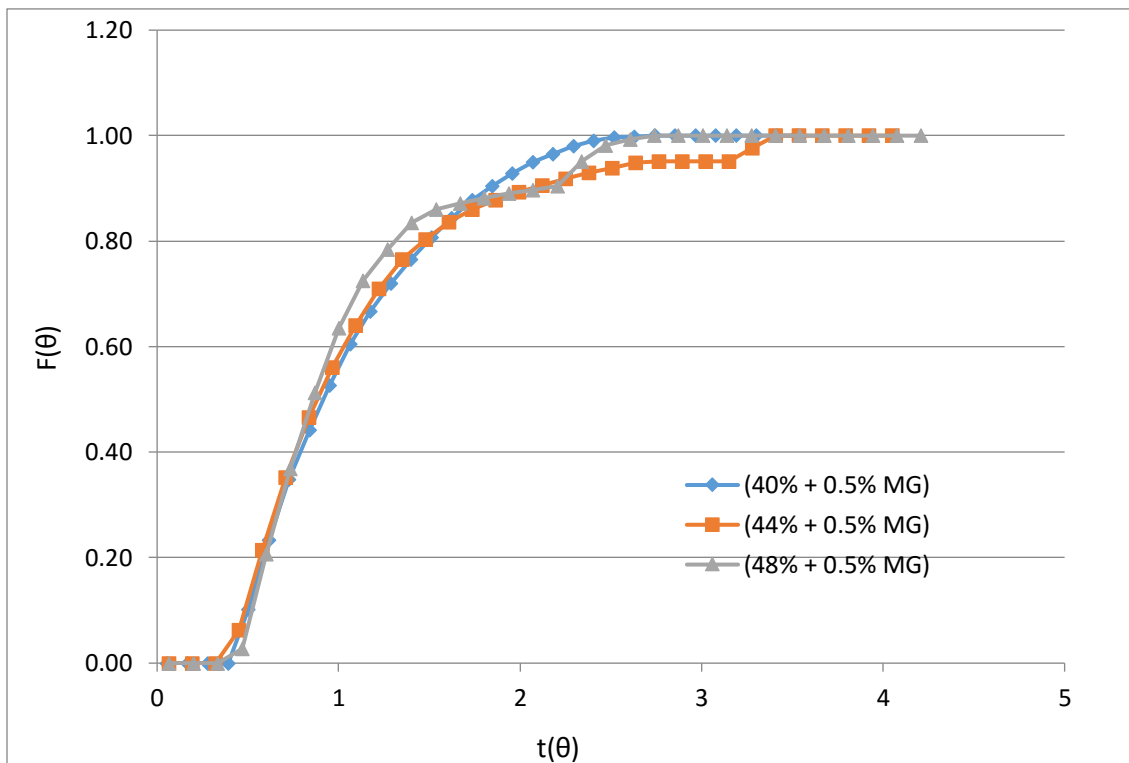
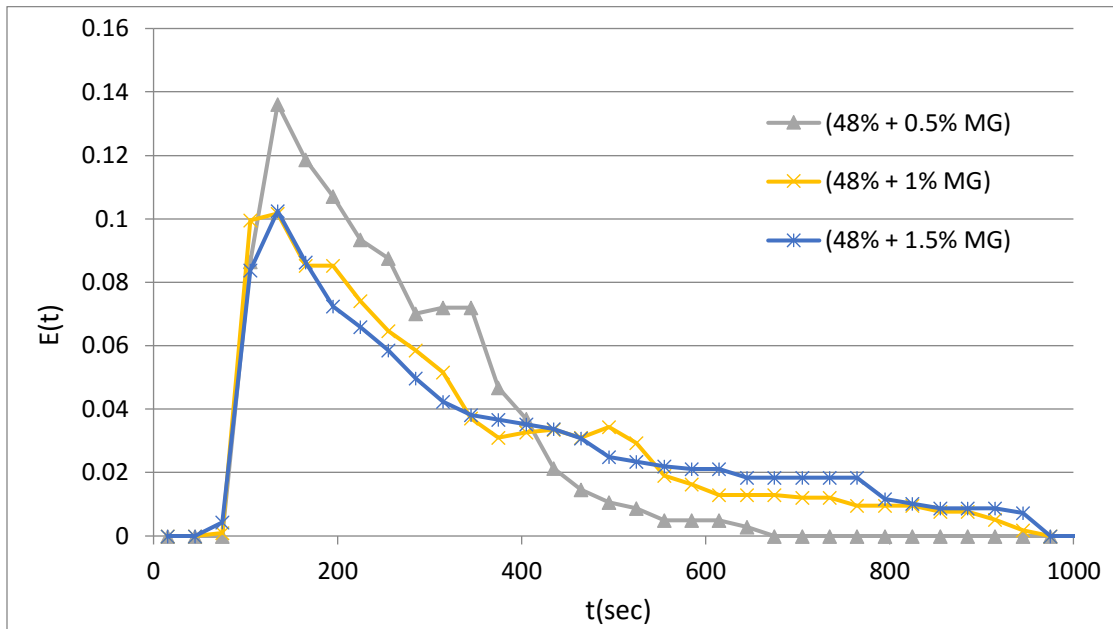
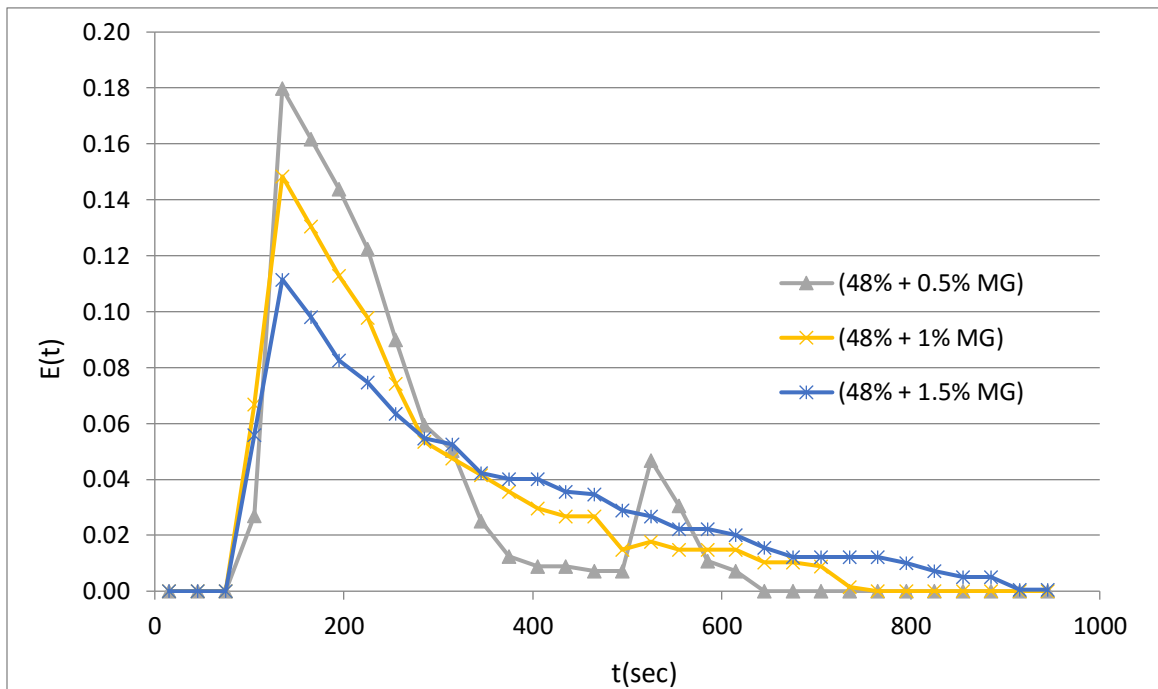


Figure 3.4: Effect of different lipid contents on E(t) and F(θ) curves.

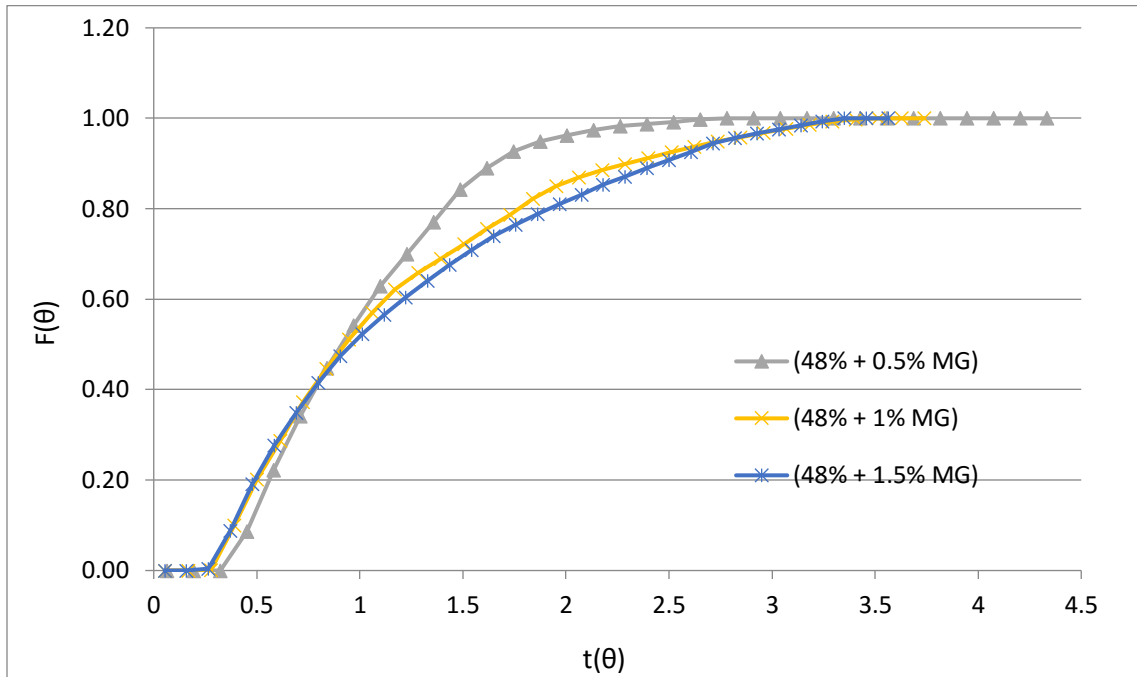
a) Effect of lipid content on E(t) curves distribution with time for first experiment.



b) Effect of lipid content on E(t) curves distribution with time for second experiment.



c) Effect of lipid content on $F(\theta)$ curves distribution with normalized time for first experiment.



d) Effect of lipid content on $F(\theta)$ curves distribution with normalized time for second experiment.

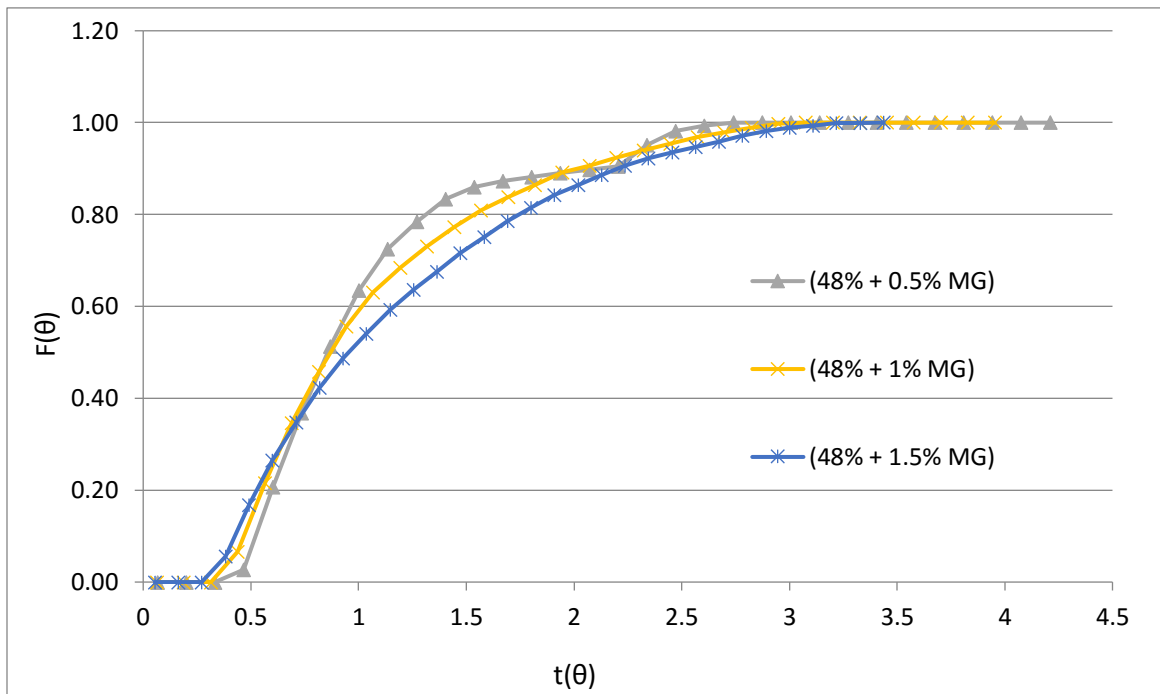
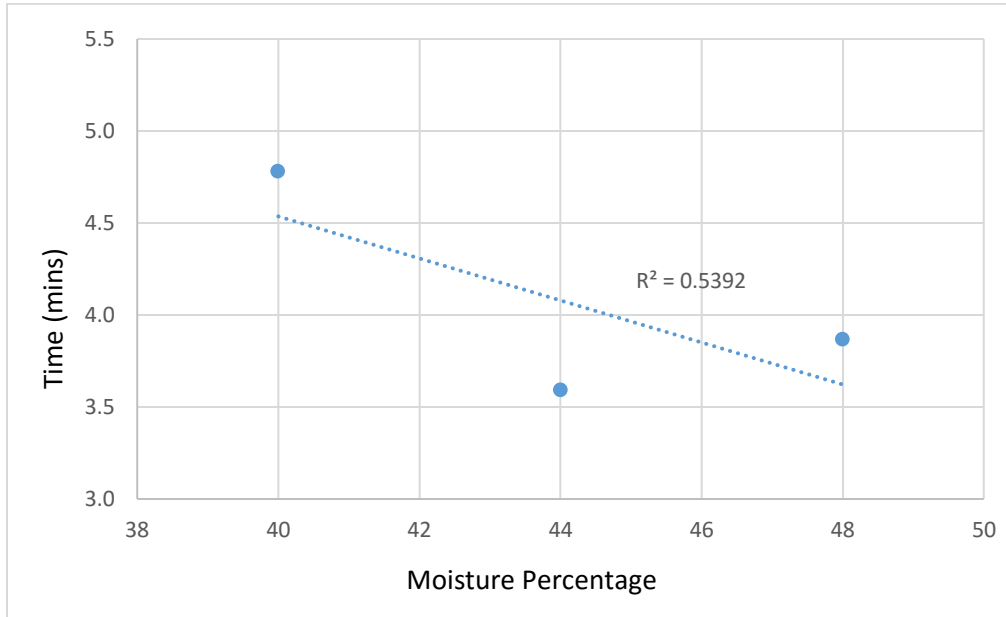
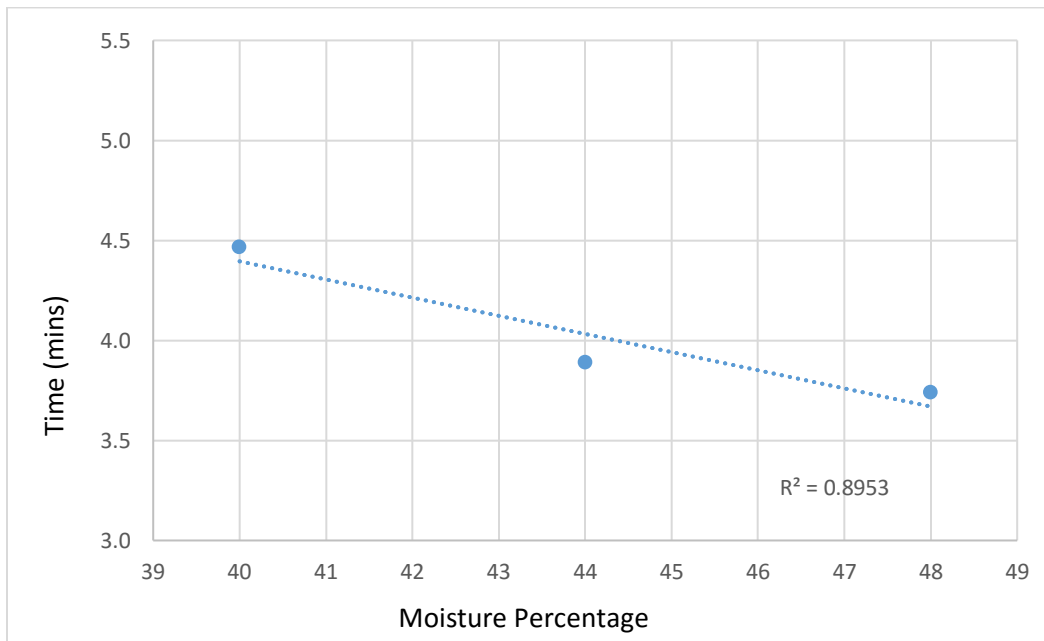


Figure 3.5: Correlation of mean residence time with different in-barrel moisture and lipid content.

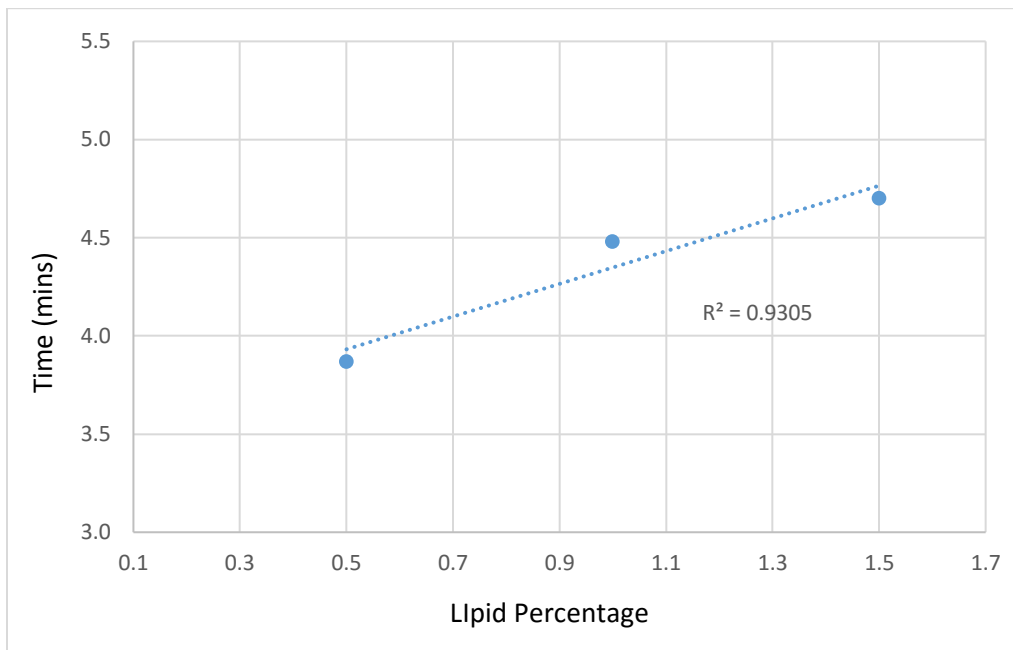
a) Correlation between moisture content and mean residence time for first experiment.



b) Correlation between moisture content and mean residence time for second experiment.



c) Correlation between lipid content and mean residence time for first experiment.



d) Correlation between lipid content and mean residence time for second experiment.

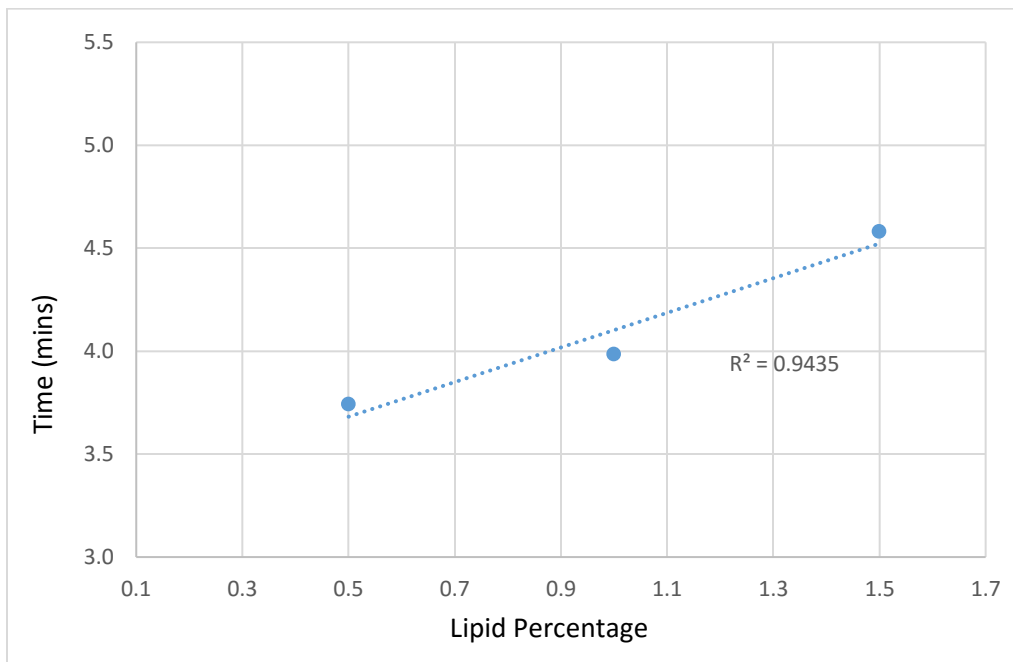
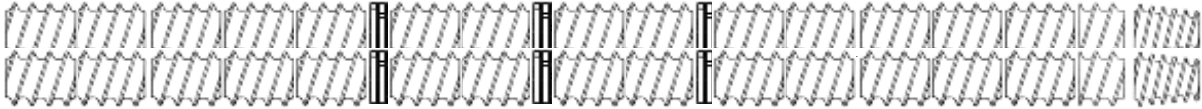


Figure 3.6: Screw profile used in residence time study.

Feed Inlet



Head 1	Head 2	Head 3	Head 4	Head 5
60°C	70°C	90°C	60°C	60°C

Chapter 4 - Quantification of process moisture and mono-glycerides levels for the development of sorghum, teff and millet precooked pasta

Abstract

Precooked pasta was prepared using sorghum at different mono-glycerides concentrations (0.5%, 1%, and 1.5%) and in-barrel moistures (40%, 44%, and 48%) in pilot scale twin screw extruder. Optimized mono-glycerides (1%) concentration and process moisture (48%) was further used to produce teff and millet pasta. The produced pastas were analyzed for cooking loss, water absorption, gelatinization, pasting and textural properties. Increase in process moisture significantly reduced solid losses and water absorption ($p < 0.05$). Higher concentrations of mono-glycerides significantly increased cooking losses and water absorption of sorghum pasta ($p < 0.05$). Sorghum pasta formulated with 1% mono-glycerides and produced at 48% moisture was of the superior quality over teff and millet pasta. Addition of mono-glycerides significantly affected pasting and textural properties ($p < 0.50$). Control wheat pasta resulted in higher firmness and teff pasta was higher in stickiness post cooking.

Introduction

Sorghum (*Sorghum Bicolor* L. Moench) is an imperative tropical crop belonging to Poaceae family that is grown in many parts of Asia, Africa, and Latin America (Anglani, 1998). Sorghum's comparative advantage is its drought tolerance; resistance to mycotoxins and fungi; and survivability in relatively adverse climatic conditions. The annual sorghum production in 2016/2017 was 58.6 million tons. Sorghum is the fifth most important crop in the world after wheat, rice, corn and barely. Sorghum outclasses other cereals under various environmental stresses and is thus generally more economical to produce. Around 35% of sorghum is now grown for human consumption. The rest of the crop is used for animal feed, alcohol and other industrial products. Sorghum (or milo) represents the third largest cereal grain in the United States. The

United States is the largest producer accounting for 20% of world production, and 57% of world sorghum exports in 2010/11 (USDA-FAS, 2012).

Starch is the main component of sorghum grain, followed by protein, other non-polysaccharides (NSP) and fat (Dicko et al., 2006). BSTIN-NRC, 1996 found that the average energy value of whole sorghum flour is 356 kcal/100g. Sorghum has maize and wheat like macromolecular composition of sorghum. Sorghum is a rich source of various phytochemicals (including phenolic compounds, plant sterols and policosanols) that are integral cellular components. These phenols act a natural barrier of plant against pests and diseases, while plant sterols and policosanols are components of wax and plant oils. The phytochemicals content of sorghum has gained increased interest around the world due to their antioxidants activity, cholesterol lowering properties and other potential health benefits. The sorghum phenols are of two major categories; phenolic acids and flavonoids.

Sorghum is used in a variety of foods for human consumption. In general, white sorghum grain processed into flour and other products such as snack, cookies and other modern foods. In United States, the use of white sorghum products is limited to consumers who are allergic to gluten. In current scenario, white sorghum is being used as a substitute to wheat products for produce gluten free products. Several other varieties of sorghum are also used for food application in Africa, China, India, Central and South America. In Eastern and southern Africa, various sorghum varieties with tannin content are extensively grown and used for the preparation of traditional staple foods, porridges and alcoholic beverages. Some African cultures particularly prefer high tannin sorghum porridges because it remains longer in stomach and farmers/ field workers feels full throughout the day. Other pigmented sorghum varieties are also preferred based on color characteristic they offer in different foods. Some of dark red color sorghum is used due to traditional health benefit phenomenon in pregnant women and are therapeutic against diseases of digestive system. However, in developed countries the use of pigmented sorghum is almost non-existent. The scientific

community needs to develop innovative ways of incorporating sorghum into mainstream food chain to utilize health benefits they offer.

The formation and presence of resistant starch in sorghum based food products impairs its digestibility in infants (FAO, 1995). The protein content ranges from 7 to 15% for sorghum grain (FAO, 1995; Beta et al., 1995). Sorghum proteins are classified solubility basis, and further divided into albumins, globulins and kafirins (aqueous-soluble prolamins), cross linked kafirins and glutelins (Jambunathan et al., 1975). Hamaker et al. (1995) and Duodu et al. (2003) found kafirins comprise about 50-70% of total proteins. α -Kafirins (23 and 25 kDa) are considered as the principle storage proteins and make up about 80% of the total kafirins. While, β -kafirins (16, 18, and 20 kDa), and γ -kafirin (28 kDa) comprise about 5% and 15% of total kafirins.

The presence of protease resistant reduces sorghum protein digestibility (Oria et al., 1995; Anglani, 1998). Axtell et al. (1981), Taylor and Taylor (2002) have shown decreased protein digestibility upon cooking. The reason for low protein digestibility is the interaction between protein-protein, protein-carbohydrate, protein-(poly) phenol and carbohydrate-(poly) phenol (Axtell, 1981; Taylor and Taylor, 2002). Sorghum grain is a rich source of more than 20 essential minerals (BSTID-NRC 1996), like iron, zinc, potassium and phosphorous (Glew et al., 1997; Anglani, 1998). It is also naturally enriched with various Vitamin-B compounds such as thiamin, riboflavin, vitamin B6, biotin and niacin (Hegedus et al., 1985). Sorghum is storehouse of nutrients, minerals and vitamins but still sorghum products are nutritionally deficient and organoleptically inferior. The possible reason could be anti-nutritional factors (ANF) such as tannin, phytic acid, and polyphenol and trypsin inhibitors. These anti-nutritional binds vitamins, proteins, mineral in food complexes making them unavailable for human nutrition (Elsheik et al., 2000; Gilani et al., 2005; Idris et al., 2007). Yoon et al. (1983), Knuckles et al. (1998) and Mohammed et al. (2011) found that the presence of anti-nutritional factors limits digestibility of proteins and carbohydrates by inhibiting proteolytic and amylolytic enzymes. All these qualities make sorghum a very healthy functional grain. The objectives of this study were to engineer a white sorghum precooked pasta based and compare with other non-traditional grains such as teff and millet.

Teff (*ivory*)

Teff (*Eragrostis tef*) an ancient grain from the lands of Ethiopia is on the smallest grain on earth (Adebowale et al., 2011). There are several varieties of teff available from dark red to white teff grain; nutritional properties of teff vary according to type of teff. In this research study, we have white (*ivory*) teff grain to produce precooked pasta formulated with salt, starch and mono-glycerides (MG). The carbohydrate content of teff is close to 80%, teff starch granules are very small 2.6 μm with 20-26% amylose content (Bultosa, 2007). The average crude protein of teff is in the range of 8 to 11 percent, teff is a store of amino acids such as glutamine, alanine, leucine, and proline (Adebowale et al., 2011). Red teff has a higher iron and calcium content than mixed or white teff (Abebe et al., 2007). Teff is a protein and iron rich grain; a rich iron source can be used as good alternative (Adish et al., 1999). Alaunyte et al., (2012) found that iron content of wheat reached more than double with 30% teff flour supplementation. The smaller starch granules size and large surface area are more susceptible to enzymatic attack (Tester et al., 2004). Wolter et al. (2013) reported glycemic index of teff (74) significantly lower than that of white wheat (100) but comparable to that of sorghum (72) and oats (71). A detailed profile of teff is explained earlier in chapter 1.

Millet (*proso*)

Millet is a type of cereal usually grown in Asian and African countries and some parts of Europe. They are consumed as a staple food by most people of arid and semi-arid tropics of the world. Proso millets (*Panicum miliaceum* L.) are the oldest cultivated millet crops and are majorly cultivated in harsh conditions as an alternative to maize because of their better adaptability to arid and barren lands than other crops (Panaud, 2006). Millets are gaining importance because they offer several nutraceuticals, minerals and vitamins to the people with low purchasing power (Taylor and Emmanbux, 2008). Proso millet has excellent nutritional properties and can become a basic resource for food diversification (Young et al., 2010). Proso millet is called millet, hog millet and yellow hog can be used in many different fields such as food, feed and functional ingredients. (Badau et al., 2005) used ground millet flour to bake flatbreads and brew beer. The

proso millet is a rich source of protein, minerals, and vitamins, and its nutritive parameters are comparable or better than common cereals. Furthermore, the quantities of nutrients in proso millet are very like to the recommended ratio of protein, saccharide, and lipids (Kalinova and Moudry, 2006). Proso millets protein has a valuable influence on metabolism of cholesterol and has been reported to raise the plasma high-density lipoprotein (HDL) without increasing the low-density lipoprotein (LDL) levels (Nishizawa and Fudamoto, 1995). It is another preventive food for liver injury (Nishizawa et al., 2002) and is a suitable foodstuff for patients requiring gluten-free diet (de la Barca et al., 2010).

Sorghum, teff and millet flour was used independently to enrich non-gluten precooked pasta. Gluten-free (GF) foods are typically based on rice and maize, and have a comparatively low content of quality proteins, and are low in fiber, calcium, and iron. GF products also have a high fat and caloric content, to compensate for decreased sensorial acceptability (Thompson, 2009). Micronutrient content of teff and millet is higher than wheat and corn, whereas as sorghum offers several health benefits such as has lower glycemic index, high protein and antioxidants.

To produce high quality GF pasta requires additives or processing techniques to modify in a suitable way the properties of macromolecular components (starch and proteins) relevant to the structure of the final product (Lai, 2001). Pagani (1986) improved rice pasta textural properties by improving gelatinization or steaming of pasta. Extrusion cooking process was developed for the starting flour was followed by conventional pasta-making process to produce rice pasta (Marti et al., 2010). Extrusion-cooking causes starch gelatinization followed by retro-gradation, forming a rigid starch network and improving the cooking quality of the product.

The goal of this work was to prepare high quality precooked pasta based on sorghum, teff and millet flour. The finished product was analyzed for various quality attributes such as cooking loss, water absorption, gelatinization, rapid viscosity analysis and texture profiles of both cooked and uncooked pasta.

Materials and Methods

Raw Materials

Durum wheat semolina was donated by durum processing and milling operation, Tree house, MO. White decorticated coarse sorghum flour with average particle size of 125 μ m of lot number KSU-170330-09 was purchased from Nulife Market, Scott city, Kansas. Ivory teff flour was obtained from “Maksal Teff”, The Teff company, Idaho, lot number 1801607 (MFD:03/08/2017). Distilled mono-glycerides DIMODAN HS K-A was obtained from Danisco USA Inc., New Century, Kansas USA Lot number 1142945586, product no 810773. Iodized salt was purchased from Morton salt Inc., Chicago IL Lot number 23960049. Hulled proso millets were obtained from Red River commodities USA, Lot number 16128FC. Millets were further ground into flour by using hammer mill, Schutte Buffalo Hammer mill (Buffalo, NY, USA) fitted with 0.23 mm (230 μ m) screen. The milled flour was collected in a tub and cooled to room temperature. The cooled flour was packed directly into 50 lb, three layered paper bags and sealed till further use. Coarse sorghum flour was obtained by hammer milling of sorghum meal (Lot# 1835-170217-27) purchased from Nulife Market, Scott city, Kansas. A screen of size 510 μ m was used to obtain targeted flour particle size of coarse sorghum flour. Three different flours, salt and mono-glycerides (MG) were blended in appropriate rations – (98.5% flour, 1% salt and 0.5% MG), (98% flour, 1% salt and 1% MG), and (97.5% flour, 1% salt and 1.5% MG) using a ribbon blender and mixed for 5 minutes. The blends were mixed in batches of 176 pounds. The blends were collected in multi-layered paper bags from bottom of the mixer by opening a sliding door.

Experimental Design

Three different grains flours sorghum, teff and milled were used as a base material to process precooked pasta in a twin-screw extruder. The first experiment was design to optimize sorghum pasta process moisture conditions (40%, 44% and 48%) at three different MG levels (0.5%, 1% and 1.5%). Sorghum flour with two different particle sizes (125 and <510 μ m) was also used. The final product was compared with durum wheat

semolina particle size (238 μm) pasta obtained as lab control.

The optimized conditions of sorghum pasta were used to run second experiment, where durum semolina, sorghum, teff and millet was processed at same conditions. The optimized MG level was 1% and process moisture content was 48%. Teff and millet were additionally deigned for treatments with 10% native corn starch formulations. Table 4.1 and 4.2 representing experiment designs used for this study.

Extrusion Process

The blends of flour, salt and MG were processed in a pilot scale co-rotating twin screw food extruder X-52 (Wenger Manufacturing Inc., Sabetha, KS, USA) equipped with a differential diameter cylinder preconditioner (DDC2, Wenger Manufacturing Inc., Sabetha, KS, USA). A five-independent zoned 1326 mm long barrel fitted with 52 mm screw diameter screws with an L/D ratio of 25.5:1 was equipped in extruder assembly. The barrel temperatures were increased to 60°C in zone-1, 70°C in zone-2, 90°C in zone-3, 50°C in zone-4, and 50 in zone-5. The heating of jacketed barrel was controlled by oil and heating elements. A loss in weight single screw feeder (Wenger Manufacturing Inc., Sabetha, KS, USA) was adjusted to feed 70 kg per hour into preconditioner. Steam and water were added into preconditioner, and the amount of steam added into preconditioner was ranged between 19.4-21.9 kg/h and water was 5.3-7.7 kg/h. The steam flow into preconditioner was controlled by Digital Pressure High Accuracy Resonate Pressure (DPHARP) sensor, model-EJA110A, style-S1, No-JEJAUR683, make YOKOGAWA. The water flow rate into preconditioner was controlled by remote flow transmitter, model RFT97121PNUR, RFT9712 S/N 59140 and mass flow sensor model-DS012H205SUP, sensor S/N 179066, and meter type-21; make Micro Motion Inc., CO, USA. Preconditioner discharge temperature was always maintained above 85°C (maximum was 93°C) with the help of right combination of water and steam in the preconditioner. Screw speed of differential diameter cylinder preconditioner was kept constant at 305 RPM.

Process moisture content of feed mix was adjusted to 40%, 44% and 48% (wet basis) based on initial moisture content of blends and additional water injected into extruder barrel to achieve target moisture levels. Extruder water flow rate was also controlled by remote flow transmitter model-RFT97121PRU, RFT9712 S/N 70307, SEN S/N 203177 and mass flow sensor model-DS012S100SU, meter type-1, Micro Motion Inc., CO, USA. Steam flow rate into extruder ranged from 7.5 to 9.0 kg/h, was controlled by DPHARP sensor, model-EJA110A, style-S1, No-JEJAUR682, make YOKOGAWA. Screw speed of extruder was kept constant at 252 RPM. Thermocouples mounted at each zone of extruder barrel were used to record respective (set and actual) zone temperatures from control panel. Die temperature and pressure was also recorded directly using thermocouple and pressure gauge respectively. Rotini pasta die, make Maldari, Number- 47637, die head number 55376-1 with 19 (insert) face openings was attached at the exit end of barrel. The die open area was 0.01796 square inch per insert. A two-blade air pressure (75 PSI) rotating face cutter was used to cut rotini shaped pasta. The cut pasta pieces were pneumatically conveyed to Wenger Double Pass Dryer/Cooler (Series 4800, Wenger Manufacturing Inc., Sabetha, KS, USA) operating at 71°C. The total retention time in dryer was 59 mins (8 mins for top and 40 minutes for bottom belts). Cooling was accomplished by room temperature air with 11mins retention time on cooling belt.

Specific mechanical energy (SME) for each treatment was calculated as follows:

$$SME = \frac{\frac{(\tau - \tau_0)}{100} \times P_{rated} \times \frac{N}{N_{rated}}}{\dot{m}}$$

τ = operating torque (%); τ_0 = no-load torque (%); P_{rated} = rated power (37.3 kW), N = screw speed (rpm); N_{rated} = rated screw speed (336 rpm), and \dot{m} = net mass flow rate of pasta at die exit (kg/s).

Cooking Process

Optimum cooking time for precooked pasta was the time required for opaque central core of the pasta to disappear when squeezed gently between two glass plates, AACC Approved Method 66–50 (AACC, 2000). This scientific approach was used to determine optimal cooking time for durum semolina, sorghum, teff and millet. An optimal cooking time of 5 min 36 secs was for durum pasta, 4 mins 42 secs for sorghum, 4 mins 36 secs for teff and 3 mins 46 secs for millet. Commercial wheat and rice pasta control was cooked as per cooking instructions which was 10 to 12 mins. An optimal cooking time of 5mins for all pasta variants was used for determination of cooking loss, water absorption/ weight gain, firmness and stickiness.

Cooking Loss

Dry/ solid matter losses during cooking were determined by AACC Approved Method 66–50 (AACC, 2000). Weighed pasta samples (25g) were cooked to optimum time in 300 mL of distilled water in a beaker, rinsed in a stream of cold water for 30 secs and drained for 2 mins. Cooked pasta and rinse water were collected, and volume made to 500 mL. The beakers carrying liquid were evaporated to dryness (constant weight) in air oven at $100\pm 1^\circ\text{C}$ for approximately 24h but may vary with oven capacity, load, etc. After complete dry beakers were taken out from oven, and cooled in desiccator to room temperature and weighed. Duplicate samples were carried out for precise results. Formula used to calculate cooking loss is as follows:

$$\text{Cooking loss} = \frac{\text{Dried weight of residue}}{\text{Weight of sample}} \times 100$$

Water Absorption capacity

Water absorption or water uptake is the amount of water absorbed or retained by pasta. It is related to functionality parameters such as cooking time and texture following cooking. Water uptake in pasta products is one the most important parameter post

cooking which also affects weight gain and textural properties. It can be measured during cooking or solid loss analysis. Pasta samples (25g) were cooked to optimum time in 300 mL of distilled water in a beaker, rinsed in a stream of cold water for 30 secs and drained for 2 mins. The drained pasta weight was measured for water uptake. All the samples were subjected to duplicate for precise data. Formula used to calculate water absorption/uptake is as follows.

$$\text{Water absorption \%} = \frac{(\text{Cooked product weight} - \text{Dry product weight})}{\text{Dry product weight}} \times 100$$

Thermal Analysis-Differential Scanning Calorimetry

To comprehend physical transformation of starch and proteins termed as starch gelatinization and protein denaturation, a calorimetric measurement was carried out for each raw material blend and extruded products. A differential scanning calorimeter (DSC) instrument Q100 DSC (TA Instruments, New Castle, DE, USA) was used to for analysis. A sample of 8-10 mg was weighed into large volume stainless steel DSC pans (Part no.03190029, Perkin Elmer Health Sciences Inc., Shelton, CT, USA). Distilled water was added to samples in the pan to obtain a solid to water ratio of 1:2 (Stevens and Elton, 1971; Zhu et al., 2010). To prevent water leakage pans were hermetically sealed and samples allowed to equilibrate overnight. The instrument was calibrated using indium as reference material. An empty sealed pan was used as reference for all experiments.

The program used consisted of several steps which includes both heating and cooling steps is as follows. First, is to equilibrate at 10°C, heating pans from 10°C to 140°C the at rate of 10°C/min, mark end of cycle 1, cooling down the sample from 140°C to 10°C at the rate of 25°C/min, mark end of cycle 2 with nitrogen gas flow rate of 50mL/min. The samples were again rescanned with heating from 10°C to 140°C at the rate of 10°C as final phase of the test; mark end of cycle 3. DSC data for each gelatinization and denaturation endotherm was analyzed for transition temperatures, onset (T_o), peak (T_p),

and endpoint or completion (T_c) and enthalpy (ΔH) using TA Instruments Universal Analysis Software (version 5.4.0). All reported data were subjected to duplicates.

Total cook (%) was calculated as a ratio of total enthalpy transition difference which includes transition enthalpies for starch and protein fractions of binary blends. It is represented as below:

$$\text{Total cook \%} = \frac{\Delta HT_{\text{raw}} - \Delta HT_{\text{extruded}}}{\Delta HT_{\text{raw}}} \times 100$$

Where, ΔHT_{raw} = Total enthalpy of transition of raw binary blend,

$\Delta HT_{\text{extruded}}$ = Total enthalpy of transition of extruded binary blend

Pasting Properties

Rapid Visco Analyzer (RVA) provides an index of how cooked a sample is by re-cooking under relatively low shear and in excess water and measuring pasting viscosity throughout test. Pasting properties of each flour and blends were examined using RVA. (RVA 4, Newport Scientific Pvt. Ltd., Warriewood, NSW, Australia). The RVA interface was equipped with software – Thermocline for Windows (version 3.15.2.298) for controlling test and analyzing results post-test. The sample size was 3.5 to 4.0 g and amount of water added was from 24.0 to 25.0 ml (corrected for 14% moisture basis). Pasting properties were determined after running samples on standard AACC profile (AACC 76-21.01, 1999) with a run time of 13 minutes. The temperature range was fixed between set points; rate of heating and cooling was 12°C/min. The paddle speed was set at 960 rpm for first 10 secs then 160 rpm for remaining test time. Peak viscosity (PV), pasting temperature (PT), trough viscosity (TV), breakdown (BD), final viscosity (FV) and setback (SB) were recorded. The viscosity values were measured in cP (centipoise) units. All measurements were performed in duplicate for both raw material and extruded products.

Texture Profile Analysis

The texture properties of pasta after cooking are extremely vital macroscopic chemical-physical and sensory properties for assessing quality of pasta. They signify some of the features that consumers are more observant during consumption. Pasta textural properties were evaluated using a TA.XT plus texture analyzer system (Stable Micro Systems, Surrey, UK).

The flexural strength or breaking strength/stress of uncooked (rigid) samples was determined by performing a three-point bend test. Uncooked pasta samples approximately 5 cm in length were placed on fixture attached to the instrument. The fixture was placed to supports samples across a span to hold sample horizontally across test probe (TA46 blind edge). A force (5.0 gm) was applied to the center of sample (which is also central to the supports) and breaking stress was determined. The breaking strength (force per unit width) or breaking stress (force per unit area) of sample is taken as the maximum strength or stress value of the curve. The distance to break gives an indication of brittleness of sample as this show how far a sample can be deformed before fracture. The gradient of slope indicates sample toughness; the higher the gradient the tougher the sample. The test parameters set were as follows: test mode= compression; pretest speed= 1.0 mm/sec; test speed= 1.0 mm; posttest speed=10.0 mm/sec; target mode = distance; distance= 15.0 mm; trigger type=auto-force; trigger force= 5.0 gm.

Firmness of cooked noodles was measured by AACC Approved Method 66–50 (AACC, 1999) for pasta with modifications. Firmness or two bite test is defined as the maximum force at first compression (first peak force). Stickiness is defined as the maximum negative peak force to separate the probe from sample surface upon retraction (the higher the negative force values the sticker is the sample). The test parameters set for the test were as follows: test mode= compression; pretest speed= 1.0 mm/sec; test speed= 1.0 mm; posttest speed=1.0 mm/sec; target mode = strain; strain= 85%; count =2; trigger type=auto-force; trigger force= 5.0 gm. A 2" diameter and 20 mm tall cylindrical aluminum probe was used to perform the test. To manage the standard deviations, results were obtained after taking the average of 25 measurements.

Statistical Analysis

All the results were analyzed using analysis of variance (ANOVA) with general linear model procedure (SAS version 9.1, SAS Institute, Cary, North Carolina, USA). When significant effects ($p \leq 0.05$) were indicated by ANOVA, Tukey pairwise comparisons were conducted to distinguish which treatments differed significantly ($p \leq 0.05$). Pearson Correlations was used to establish correlation values.

Results and Discussion

Pasta Production

The process parameters of experiment are summarized in Table 4.3, 4.4 and 4.5; indicating process moisture, post drying product moisture and SME. The process was designed to run moisture ranged from 39.5% to 47.9% meeting study objectives of low to high process moisture contents. The dried moisture of pasta was from 9.80% to 12.23%. The moisture content of dried pasta ranged from 9.8% to 12.23%, highest was for wheat pasta and lowest for millet pasta. Sorghum, millet and teff pasta were dried at same conditions, for dried sorghum pasta the moisture content ranged from 10.36% to 11.77%, teff pasta 10.83% to 11.05% and millet pasta 9.80% to 10.07%. The final moisture was similar to of commercial pasta Barilla Wheat Rotini (11.13%) and Barilla Penne rice pasta (10.56%).

The calculated values of SME consumed during the experiment were represented in Table 4.3, 4.4, 4.5. The SME values ranged from 43 to 162 kilo joules per kilogram. Among three grains at same processed at conditions teff pasta took highest energy 80 kJ/kg, followed by sorghum 64.3 kJ/kg and millet 57.8 kJ/kg. The teff starch granules are densely packed require higher energy to cook (Bultosa, 2007; Bultosa et al., 2002). The higher lipid content of millets has reduced shear and caused lubricating affect which lowered energy required in extruder (Ravindran, 1991; Kalinova, 2002). Gour and Gautam (2003) found drop in SME input by increasing fat levels in twin screw extrusion of rice flour.

Sorghum pasta was developed at three different levels of moisture (39%, 44% and 48%). The SME input increase with reducing in process moisture levels and vice versa (Figure 4.10). Treatment with 39% moisture content consumed highest energy (162.3 kJ/kg); followed by 44% (91.2 kJ/kg); and 48% (56.2 kJ/kg). In extrusion process water works as a plasticizer. The plasticizing effect of water lowers the viscosity of the melt, resulted in lower SME. Akdogan (1996) also reported significant increase ($p < 0.05$) in SME with decrease in moisture. Decline in SME with increasing moisture agrees with findings of Van Lengerich (1984), Van Zuilichem et al. (1975), Faubion et al. (1982), Bhattacharya and Hanna (1987), and Likimani et al. (1991) although moisture conditions used in these studies were lower. The second part of the experiment was three different levels (0.5%, 1% and 1.5%) of MG (Table 4.4). There was no significant difference in SME input at different levels mono-glycerides in formulations.

Cooking Loss

Several phenomena are occurring during cooking such as hydration, starch gelatinization and interaction with non-starchy matrices. The optimal cooking time of sorghum, teff and millet pasta was different. The differences between cooking time could be attributed to different gelatinization temperatures of starches (Singh, Singh et al., 2002). All pasta samples were cooked for 5.0 mins to measure cooking losses, Cooking loss values were lower than 10% of pasta mass which indicates good quality of precooked pasta (Kim et al., 1996; Wang et al., 1999). Cooking losses sorghum, teff and millet pasta were comparable to those of wheat pasta, which was used as control (Table 4.5). Cooking losses of only sorghum pasta processed at different moisture and MG levels ranged from 4.0% to 6.8% (Table 4.3 and 4.4). The lowest was for treatment formulated with 0.5% MG processed at 48% moisture content and highest at 44% moisture. Cooking loss values were significantly different ($p < 0.05$). Use of food emulsifier (MG) in blends resulted in higher cooking losses (Figure 4.1b). Treatment with 0.5% MG had 4% cooking loss; 1% MG was 5.22%; 1.5% MG was 5.85% (Table 4.4). The high cooking losses were due to higher water solubility of sorghum flour, results shown in Table 4.3 and 4.4. Increase in MG results in higher cook weight (water

absorption) (Table 4.4). A 0.5% increase in MG in sorghum resulted in significant weight gain post. This may be due to the ability of MG to form complexes with amylose which prevents the starch granules from gelatinization. The uncooked sorghum starch might have leached out fast and resulted in higher cooking loss. Similar results were reported by Chapen et al. (2008) for extruded rice vermicelli formulated with MG. A negative effect of MG on cooking loss of sorghum pasta was observed. Treatment with larger particle size resulted in higher cooking loss (6.43%). Sorghum pasta yielded lowest cooking loss (5.24%), teff pasta (7.40%) and millet pasta (7.26%) processed at same moisture (48%) and MG (1%), (Figure 4.1c). Addition of corn starch lowered cooking loss by 27.2% for teff but increased 13.1% for millet.

Water Absorption

Hydration of cooked pasta is an indicator of water uptake during cooking, and relates to various macroscopic molecular changes takes place in starch and protein bodies (Sozer et al., 2007). Water absorption (WA) values of sorghum, teff and millet pasta are represented in (Table 4.5). The WA of cooked pasta was significantly affected by process water and MG levels ($p < 0.05$) (Table 4.3 and 4.4). Ding et al. (2006) also reported significant drop in water absorption index by increase in feed moisture of expanded snack in twin screw extruder. The range of WA values was from 125.86% to 244%; highest was for millet formulated with 1% MG and processed at 48% moisture. Millet pasta disintegrated rapidly during cooking which increased the surface area of pasta resulted in higher water absorption. The low gelatinization percentage could be the possible reason for rapid disintegration of millet pasta (Table 4.6). A trend of decrease in hydration with increase in process moisture was observed in sorghum pasta treatments ($R^2 = -0.99$). Sorghum processed at 40% moisture gained highest weight over 44% and 48% (Figure 4.2a).

The WAI measures the amount of water absorbed by starch and can be used as an index of gelatinization (Anderson et al., 1969). The conversion of raw starch to a cooked and digestible material by the application of water and heat is one of the important effects that extrusion has on the starch component of foods. It may be expected that as starch granule structure is disrupted more water is bound to starch molecule resulting in

higher WA. The higher SME inputs generates more shear which lead to complete disruption of protein networks and starch granules, resulted in higher penetration of water during cooking (Mercier and Fillet, 1975). The increased accessibility to the protein polar amino-acid groups during cooking because of the denaturation of proteins, especially albumins, could have enhance the affinity for water (Alonso et al., 2000) thus resulting in greater WA.

The WA of cooked pasta was found to increase with increasing levels of MG for sorghum pasta ($R^2=0.96$) (Figure 4.4a). Sorghum pasta with 0.5% MG gained weight by 126%; 1% MG- 170%, 1.5% MG- 191% (Figure 4.2b). The addition of MG inhibits starch swelling and lowers gelatinization of starch granules, for such starch granules most of the water absorption takes place during secondary cooking (Donnelly and Ponte, 2000; Eliasson and Krog, 1985). Chapen et al. (2008) reported increase in spaghetti hydration formulated with higher MG percentages. Sorghum pasta with 1% MG gained maximum weight with lowest solid loss over wheat teff and millet (Figure 4.2c). Giuberti et al. (2015) reported high WA index of spaghettis made from non-wheat flours. Adding native corn starch into teff and millet pasta decreased hydration.

Thermal Analysis-Differential Scanning Calorimetry

Gelatinization represents the degree of cook or degree of starch transformation in any cereal based cooked product (Patton and Sprat, 1981). Gelatinization temperature represents the melting of crystalline structure of starch granules, loss of birefringence and other transformations. The medium temperature, high moisture, and relative long residence time made it a gentle cooking process for cereal-based pasta. Raw flour blends were scanned to determine onset temperature, peak temperature, and completion temperature and gelatinization enthalpy (Table 4.6 and 4.7). The gelatinization temperatures were not significantly different ($p>0.05$). The highest enthalpy input was observed for control wheat blend, followed by teff, sorghum and millet blends was the lowest (Table 4.7). Gelatinization enthalpy values were significantly different ($p<0.05$).

It was observed that starch gelatinization of all pasta formulations post extrusion ranged from 92.7% to 100% (Table 4.6 and 4.7). The lowest degree of gelatinization was for teff with 1% MG (92.7%) followed by teff with 10% starch and 1%MG (93.4%), sorghum with 1% MG (96.0%), millet with 1% MG (96.9%) and millet with 10% starch and 1% MG (97.7%), (Table 4.7). In sorghum pasta, increase in MG levels have decrease the gelatinization percentages ($R^2=0.75$) (Figure 4.5a), these values were statically different ($p<0.05$) (Table 4.6). Both teff and millet formulated with 1% MG remained lower in cooking percentages (Table 4.7). The presence of fiber and fat in whole grain teff and millet formulations cause a reduction in SME (Table 4.5) and thus led to less cook (Table 4.7). Feng and Lee (2014) reported that in extrusion lipids work as a lubricant led to lowering SME input. Lin et al. (1997) also observed that increase in fat content in extrudates decreased the degree of starch gelatinization.

Pasting Properties

Pasting properties of starch is the phenomena involving granular swelling, exudation of molecular components from granules and eventually, total disruption of granules (Atwell et al., 1988). Offline pasting viscometer was used to measure the degree of cook in starch gelatinization (Harper, 1994). Rapid Visco Analyzer (RVA) was used for offline control to measure relative starch degradation (Ryu et al., 1993). The RVA method involves re-cooking and monitoring viscosity changes. This has been used to quantify cold-swelling 'cooked' component, the 'raw' component that pastes during test, and the overall viscosity that indicates degree of starch dextrinization. Pasting properties depends upon variety and source of starch (Lineback, 1984). Pasting properties of raw flour blends are represented in Table 4.8. Sorghum flour blends show highest peak viscosity (S+MG 0.5%) 2891 ± 99 , followed by control heat 2767 ± 13 , sorghum coarse flour blend (SC+MG 0.5%) 2397 ± 19 , teff (T+MG 1%) 2240 ± 45 , and millet (M+MG 1%) 2209 ± 20 . Raw flour treatment's peak viscosity values were significantly different ($p<0.05$). Pasting temperature of raw flours blends were significantly different ($p<0.05$). Semolina (control) displayed highest pasting temperature 88°C , followed by coarse

sorghum flour 83.8°C, teff– 79.6°C, sorghum- 78.7°C and millet- 76.8°C. Likewise, the pasting time values also displayed similar trend, and were significantly different ($p < 0.05$). Sorghum coarse flour found to be having higher pasting temperature and pasting time than sorghum flour, the possible reason could be the larger particle size of coarse flour. Larger particles require more time for water and heat to penetrate into the granule, slow and delayed swelling of starch granules delays breakdown viscosity.

Millet blend showed the highest breakdown in viscosity- 1273 cP, followed by sorghum- 1004 cP, control semolina 675 cP and teff- 647 cP. All breakdown viscosity values were significantly different ($p < 0.05$). Highest setback was observed in sorghum blends, followed control semolina, millet and teff. Final viscosity and set back represents the tendency of rapid retro gradation of starch granules, sorghum blends shown highest set back followed by control semolina, millets and teff. Similar trends were observed for final viscosity. Different individual flours were subjected to RVA test without any blending's, the addition of salt and MG affected the pasting prosperities but trends remained same in raw blends (values not shown).

Pasting properties of extruded pasta formulated with three different flours are represented in Table 4.10. For sorghum variants, increase in process moisture increased peak time significantly ($p < 0.05$) (Table 4.9), but highest peak time was observed for control semolina. Decorticated coarse sorghum pasta took less time to reach peak than decorticated fine sorghum flour (Table 4.9). Both these properties were directly affected by the SME input (Table 4.4), higher energy input let to lower peak time and cold swelling. Higher SME caused greater starch damage and starch gelatinization resulted higher particle hydration at ambient temperature (Ozcan and Jackson, 2005; Whalen et al., 1997). Rapid granular swelling with high water absorption rates leading to starch solubilization and leaching of amylose below 90°C could have possibly increased viscosity. Further increase in temperature and mechanical stress during the holding phase causes further disruption of the starch granule and the remaining amylose is leaches out (Ragae and Abdel, 2006). Thus, sorghum treatments processed at low process moisture levels had significantly lower peak time and lower final viscosity.

These observations agree with Mahasukhonthachat et al. (2010) reported in the effects of extrusion kinetics on pasting properties. Among different grains teff had significantly ($p < 0.05$) higher peak time, followed by control semolina, sorghum and millet was the lowest (Table 4.10).

Peak viscosities of sorghum pasta samples were significantly ($p < 0.05$), sorghum treatments processed at lower moisture was lower peak viscosity; 40%- 464cP, 44%- 669cP, and 48%- 1718cP (Table 4.9). Decorticated coarse sorghum pasta had higher peak viscosity than decorticated fine sorghum flour. For different flour pasta's control semolina resulted in highest peak viscosity- 3847 cP, sorghum- 2612 cP, teff- 2213 cP, millet- 1152cP. The final viscosities of millet were found to be lower than that of all other grains. The presence of lipids in whole millet flour has been reported to lower the peak viscosity due to amylose-lipid complex formation (Singh et al., 2007 and Singh et al., 2003). The addition of emulsifier greatly affects the degree of gelatinization in high shear process by providing lubricating effect (Chanpen et al., 2007). The low breakdown viscosity indicates that millet starches had more resistance to high temperature and shearing and that it has higher pasting stabilities.

After the breakdown, the increase in viscosity during cooling period is indicative not only of normal inverse relationship between viscosity and temperature of suspensions but also of the tendency for swollen granules, dispersed and dissolved starch molecules to retrograde with decrease in temperature (Singh et al., 2007). The addition of MG lowered the setback viscosities in sorghum formulations (Table 4.10). The setback viscosities of control semolina were highest 5468 cP as compared to other grains, millet 5002 cP, sorghum- 3671 cP, and teff was 3478 cP. The millet starch re-absorbed water rapidly and resulted into higher setback (Table 4.10). Similar behavior of proso millet was reported by Yanez et al. (1991). The lower setback viscosity values could be due to lower extent of retro-gradation of amylose during cooling (Singh et al., 2009).

Textural properties

Three-point bend test

Hardness is the average force required for a probe to penetrate the uncooked pasta. The hardness values for uncooked pasta were observed and documented in Table 4.11 and 4.12. In Sorghum pasta variants increase in process moisture increased pasta hardness significantly ($p < 0.05$); 40%- 1.38 ± 0.22 kg, 44%- 1.75 ± 0.49 kg, 48%- 2.47 ± 0.97 kg. Hardness was positively correlated with process moisture content ($R^2 = 0.91$) (Figure 4.6a). During extrusion process, the melt swell effect and bubble growth effect both contribute to the structure change. The moisture content during extrusion processing directly affects the starch gelatinization and extrudate expansion to product (Panmanabhan and Bhattachayrya, 1989). The water acts as a plasticizer to the starch-based material reducing its viscosity and the mechanical energy dissipation in the extruder and thus the product becomes dense and bubble growth is compressed (Liu et al., 2000). This was confirmed in this study as the recorded SME inputs was found to be negatively correlated with process moisture (Table 4.3). The reduced starch conversion and compressed bubble growth would result in a dense product and reduced crispness of pasta, as observed in this work. Previous studies also reported that the hardness of extrudate increased as the feed moisture content increased (Badrie and Mellowes, 1991; Liu et al., 2000).

Results suggested that the pasta structuring agents such as MG tested in this study significantly ($p < 0.05$) affected the stress resistant properties of decorticated white sorghum pasta three-point bend test (Table 4.10). The drop-in compression force with increasing levels of MG ($R^2 = 0.89$) (Figure 4.6b). The lowering of break force for sorghum pasta samples containing different levels of MG can be attributed to two things, a) decrease in the rate of starch retro-gradation due to the formation of amylose-lipid complexes (Kaur et al., 2005), b) a weak interaction between structuring agents and sorghum flour components (protein and starch binding) induces a reduction in the stress at break values due to lower gelatinization (Chillo et al., 2009). In both the experiments control semolina pasta exhibited highest break force, followed by teff, sorghum and millet pasta (Table 4.11). Semolina pasta resulted was of highest firmness

due to strong gluten-starch network, whereas small teff starch granules tightly binds starch granules to make strong structure matrix of pasta (Bultosa et al., 2002). Higher lipids content of millets may have led to the soft texture of millet pasta (Jones et al., 1970; Ravindran,1991; Kalinova, 2002).

Firmness and Stickiness

Textural parameters especially firmness and adhesiveness are important for pasta cooking quality. The hardness values of cooked pasta were statically different ($p < 0.05$). Firmness and adhesiveness values of cooked pasta are represented in Table 4.11 and 4.12. The hardness of cooked sorghum pasta significantly increased with an increase in process moisture ($R^2 = 0.83$) (Figure 4.7a). Similarly, increase in MG percentages in the formulations resulted in lowering firmness post cooking ($R^2 = -0.89$) (Figure 4.7b). Differences in firmness values mainly arise due to the cooking of starch granules and different protein networks. Higher gelatinization of starch granules yielded into higher firmness and vice-versa. The firmness values of cooked pasta were moderately correlated with gelatinization ($R^2 = 0.54$) (Figure 4.7c). Adhesiveness or stickiness is related with the amount of starch and starch gelatinization (Matsuo and Irvine, 1970). During cooking, severe changes in the microstructure of pasta occurs. The uniformity of dry pasta starts to change by the diffusion of water from outside to the core. Closer to the surface of the pasta strand the changes are more drastic, starch granules are no longer intact as in the core and protein matrix starts to break down due to denaturation.

Firmness results of cooked pasta evaluated in this study are in agreement with Sozer et al., (2007) who reported lower firmness values of cooked spaghettis at higher gelatinization. The firmness values control pasta was highest, followed by sorghum, millet and teff (Table 4.12). The strong gluten matrix and protein network could be the possible reason for higher firmness. The inclusion of bran from whole millet flour might be the possible reason for higher hardness (Sozer et al., 2007). The firmness value of teff pasta produced on pilot scale was lower than millet pasta whereas the results were opposite in lab scale pasta. The possible reason could be the low gelatinization of teff pasta. The incomplete cooking failed to form strong starch-protein matrix of product,

which disintegrates quickly during cooking and further on textural instrument.

Stickiness figures of cooked pasta were significantly different ($p < 0.05$). The teff pasta was the stickiest 0.58 kg; control and millet 0.22 kg; sorghum pasta was least sticky- 0.12 kg. Similar, results were obtained in lab scale pasta where teff pasta had higher stickiness values over semolina and millet pasta. The likely reason that makes millets less sticky is the higher percentage of lipids than teff and sorghum.

Conclusion

In this study, twin screw extruder was successfully used to identify the optimal formulation for precooked pasta from sorghum, teff and millet flours. Cooking losses and water absorption of sorghum pasta were shown to be better, and teff and millet were comparable to the lab produced durum wheat based semolina pasta. Different process moistures and addition of different MG percentages into the raw formulation have significantly affected cooking loss, water absorption, gelatinization, viscosity properties and textural properties. Optimum process moisture and MG levels resulted in a good quality product with higher water absorption and lower cooking loss. Addition of native corn helped to improve teff pasta quality attributes but not millet flour. Higher process moisture increased the hardness of uncooked pasta, whereas addition of MG lowered hardness pasta sorghum pasta. Raw teff pasta was higher in hardness than sorghum and millet. For cooked pasta inclusion of MG lowered firmness, and teff pasta was the stickiest product. The results obtained from this study can further utilized to proliferate the use of drought resistant crops (sorghum, teff and millets) to develop human food through food processing industry and research.

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Table 4.1: Pilot scale experimental design for the optimization of sorghum pasta processed at different moisture conditions and mono-glycerides levels.

Ingredients	Control	S+MG (0.5%)	S+MG (0.5%)	S+MG (0.5%)	S+MG (1%)	S+MG (1.5%)	SC+MG (1.5%)
Semolina	98.5						
Sorghum Flour		98.5	98.5	98.5	98	97.5	
Sorghum coarse flour							98.5
Mono-glycerides	0.5	0.5	0.5	0.5	1	1.5	0.5
Salt	1	1	1	1	1	1	1
Process Moisture	46.8	44.7	39.5	47.9	47.5	47.9	47.5

*All values are represented in percentages (%) of total.

Table 4.2: Pilot scale experimental design of precooked wheat, sorghum, teff, and millet pasta.

Ingredients	Control	S+MG (1%)	T+MG (1%)	T+CS (10%)+MG (1%)	M+MG (1%)	M+CS (10%)+MG (1%)
Semolina	98.5					
Sorghum Flour		98				
Teff Flour			98	88		
Millet Flour					98	88
Corn starch				10		10
Mono-glycerides	1	1	1	1	1	1
Salt	1	1	1	1	1	1
Process Moisture	46.8	48.6	48.1	47.3	47.5	47.5

S-sorghum flour, SC-Sorghum coarse flour, control- durum semolina, T-teff flour, M-millet flour, MG-mono-glycerides, CS- corn starch.

*All values are represented in percentages (%) of total

Table 4.3: Pasta moisture, SME, WA and CL of sorghum pasta processed at different in-barrel moistures.

Treatments	Moisture		SME (%)	WA (%)	CL (%)
	Process	Dried KJ/KG			
Control	46.8	11.71±0.15 ^a	62.5	136.17±0.9 ^b	5.13±0.2 ^c
S+MG (0.5%)	39.5	10.88±0.14 ^{ba}	162.3	160.16±5.3 ^a	6.34±0.1 ^b
S+MG (0.5%)	44.7	10.49±0.0b ^{ba}	91.2	136.63±2.6 ^b	6.85±0.6 ^a
S+MG (0.5%)	47.9	11.77±0.01 ^a	56.2	125.86±2.7 ^c	4.0±0.2 ^d

Moisture in percentage, SME- Specific mechanical energy, S-sorghum flour, SC-Sorghum coarse flour, control- durum semolina, T-teff flour, M-millet flour, MG-mono-glycerides, WA-water absorption (%), CL-cooking loss (%).

Table 4.4: Pasta moisture, SME, WA and CL of sorghum pasta processed at different mono-glycerides levels.

Treatments	Moisture		SME (%)	WA (%)	CL (%)
	Process	Dried KJ/KG			
S+MG (0.5%)	47.9	11.77±0.01 ^a	56.2	125.86±2.7 ^c	4.0±0.2 ^d
S+MG (1%)	47.5	11.19±0.05 ^{ba}	57.7	169.79±2.7 ^{ba}	5.22±0.0 ^c
S+MG (1.5%)	47.9	10.36±0.07 ^b	57.0	191.28±3.4 ^a	5.85±0.4 ^b
SC+MG (1.5%)	47.5	11.11±0.03 ^{ba}	72.2	166.85±2.3 ^b	6.43±0.2 ^a

Moisture in percentage, SME- Specific mechanical energy, S-sorghum flour, SC-Sorghum coarse flour, control- durum semolina, T-teff flour, M-millet flour, MG-mono-glycerides, WA-water absorption (%), CL-cooking loss (%).

Table 4.5: Pasta moisture, SME, WA and CL of sorghum, teff and millet.

Treatments	Moisture		SME (%)	WA (%)	CL (%)
	Process	Dried KJ/KG			
Control	46.8	12.23±0.10 ^a	69.1	136.16±0.2 ^d	4.47±0.3 ^d
S+MG (1%)	48.6	10.75±0.08 ^{bc}	64.3	204.18±9.3 ^b	5.24±0.2 ^c
T+MG (1%)	48.1	10.83±0.0 ^{bc}	80.5	165.76±9.1 ^c	7.40±1.0 ^b
T+CS (10%)+MG (1%)	47.3	11.05±0.26 ^b	73.1	153.76±12.2 ^c	5.39±0.2 ^c
M+MG (1%)	47.5	10.09±0.07 ^c	57.8	244.54a±1.6 ^a	7.26±0.0 ^b
M+CS (10%)+MG (1%)	47.5	09.80±0.05 ^c	43.7	197.61±10.8 ^b	8.21±0.3 ^a

Moisture in percentage, SME- Specific mechanical energy, S-sorghum flour, SC-Sorghum coarse flour, control- durum semolina, T-teff flour, M-millet flour, MG-mono-glycerides, CS- corn starch, WA-water absorption (%), CL- cooking loss (%).

Table 4.6: Thermal characteristics of precooked sorghum pasta samples: onset (T_o), peak (T_p), completion (T_c), gelatinization enthalpies (ΔH) and degree of starch gelatinization (Δg).

Treatments	T_o (°C)	T_p (°C)	T_c (°C)	ΔH (J/g)	Δg (%)
Control	57.2±0.8 ^b	65.0±1.7 ^b	76.0±2.5 ^b	0.0±0.0 ^d	100±0.0 ^a
S+MG (0.5%)*	69.8±0.2 ^a	75.0±0.0 ^a	86.7±0.2 ^a	0.0±0.0 ^d	100±0.0 ^a
S+MG (0.5%)**	69.8±0.2 ^a	75.0±0.0 ^a	86.7±0.2 ^a	0.0±0.0 ^d	100±0.0 ^a
S+MG (0.5%)***	69.8±0.2 ^a	75.0±0.0 ^a	86.7±0.2 ^a	0.0±0.0 ^d	100±0.0 ^a
S+MG (1%)	70.8±0.8 ^a	75.8±0.7 ^a	85.9±1.4 ^a	0.0±0.0 ^d	100±0.0 ^a
S+MG (1.5%)	69.7±0.4 ^a	74.4±0.2 ^a	83.8±0.4 ^a	0.2±0.0 ^{cb}	96.0±0.8 ^b
SC+MG (1.5%)	70.5±1.2 ^a	75.7±1.2 ^a	87.5±1.4 ^a	0.3±0.0 ^b	96.0±0.0 ^b

S-sorghum flour, SC-Sorghum coarse flour, control- durum semolina, T-teff flour, M-millet flour, MG-mono-glycerides, enthalpy (ΔH) for raw semolina $\approx 9.1 \pm 0.2$, Sorghum coarse flour $\approx 10.4 \pm 0.8$, sorghum flour $\approx 8.6 \pm 1.6$, teff flour $\approx 10.2 \pm 0.2$, and millet flour $\approx 6.9 \pm 0.5$.

* At process moisture 39.5%

** At process moisture 44.7%

*** At process moisture 47.9%

Table 4.7: Thermal characteristics of precooked sorghum, millet and teff pasta samples: onset (T_o), peak (T_p), completion (T_c), gelatinization enthalpies (ΔH), and degree of starch gelatinization (Δg)

Treatments	T_o (°C)	T_p (°C)	T_c (°C)	ΔH (J/g)	Δg (%)
Control	57.2±0.8 ^b	65.0±1.7 ^b	76.0±2.5 ^b	0.0±0.0 ^d	100±0.0 ^a
S+MG (1%)	70.8±0.8 ^a	75.8±0.7 ^a	85.9±1.4 ^a	0.0±0.0 ^d	100±0.0 ^a
T+MG (1%)	70.2±0.8 ^a	77.0±0.9 ^a	88.3±0.6 ^a	0.6±0.1 ^a	92.7±0.7 ^{dc}
T+CS (10%)+MG (1%)	71.3±1.1 ^a	78.1±1.1 ^a	88.7±0.9 ^a	0.5±0.1 ^a	93.4±0.6 ^c
M+MG (1%)	70.9±0.7 ^a	75.8±0.9	85.4±0.9 ^a	0.2±0.0 ^{cb}	96.9±0.4 ^b
M+CS (10%)+MG (1%)	71.5±1.2 ^a	76.6±1.5 ^a	85.4±1.9	0.1±0.0 ^c	97.7±0.4 ^b

S-sorghum flour, SC-Sorghum coarse flour, control- durum semolina, T-teff flour, M-millet flour, MG-mono-glycerides, CS- corn starch, enthalpy (ΔH) for raw semolina $\approx 9.1 \pm 0.2$, Sorghum coarse flour $\approx 10.4 \pm 0.8$, sorghum flour $\approx 8.6 \pm 1.6$, teff flour $\approx 10.2 \pm 0.2$, and millet flour $\approx 6.9 \pm 0.5$.

Table 4.8: Pasting properties of raw pasta blends formulated with salt, MG and corn starch.

Treatments	PT _c (°C)	PT _m (mins)	PV (cP)	T (cP)	BD (cP)	SB (cP)	FV (cP)
Control	88.0±0.6 ^a	8.7±0.3 ^a	2767±13 ^{bc}	2092±167 ^a	675±180 ^{ef}	3290±117 ^c	5382±49 ^{ce}
S+MG (0.5%)	78.7±0.8 ^d	7.8±0.0 ^{bdec}	2891±99 ^{ba}	1888±73 ^{ba}	1004±26 ^{dc}	5038±19 ^b	6925±92 ^b
S+MG (1%)	77.8±0.6 ^d	7.7±0.2 ^{dec}	2863±11 ^{bac}	1859±132 ^{bac}	1004±120 ^{dc}	5205±38 ^{ba}	7064±170 ^b
S+MG (1.5%)	78.3±1.3 ^d	7.8±0.2 ^{bdec}	2836±40 ^{bc}	1997±8 ^{ba}	839±48 ^{ed}	5532±133 ^a	7529±125 ^a
SC+MG (1.5%)	83.8±0.0 ^{bc}	8.2±0.3 ^{bdac}	2397±19 ^{edf}	1960±72 ^{ba}	437±53 ^f	5575±126 ^a	7535±54 ^a
T+MG (1%)	79.6±0.8 ^d	8.2±0.1 ^{bac}	2240±45 ^{edf}	1594±15 ^c	647±30 ^{ef}	2540±214 ^d	4133±229 ^e
T+CS (10%)+MG (1%)	80.1±0.1 ^{dc}	8.4±0.0 ^{ba}	2101±88 ^{gf}	1578±37 ^{dc}	524±50 ^f	1798±230 ^{fe}	3375±192 ^f
M+MG (1%)	76.8±0.6 ^d	7.6±0.0 ^e	2209±20 ^{ef}	937±16 ^f	1273±36 ^{bac}	2857±72 ^d	3794±56 ^{ef}
M+CS (10%)+MG (1%)	79.1±2.8 ^d	7.7±0.2 ^{dec}	2440±56 ^{ed}	1104±76 ^{ef}	1336±21 ^{ba}	2758±45 ^d	3862±32 ^e

S-sorghum flour, SC-Sorghum coarse flour, control- durum semolina, T-teff flour, M-millet flour, MG-mono-glycerides, CS corn starch.

Table 4.9: Pasting properties of extruded sorghum pasta formulated with salt, MG and processed at different moistures.

Treatments	PTc (°C)	PTm (mins)	PV (cP)	TV (cP)	BD (cP)	SB (cP)	FV (cP)
Control	25±0.1 ^a	4.3±0.1 ^a	3217±67 ^a	3165±78 ^a	53±11 ^a	472±17 ^d	8637±61 ^a
S+MG (0.5%)*	25±0.1 ^a	3.2±0.2 ^c	464±00 ^e	456±10 ^d	08±10 ^c	680±28 ^d	1136±18 ^e
S+MG (0.5%)**	25±0.1 ^a	3.0±0.3 ^c	669±5 ^d	658±57 ^d	12±04 ^c	540±15 ^d	1197±42 ^e
S+MG (0.5%)***	25±0.1 ^a	3.6±0.3 ^b	1718±40 ^c	1718±40 ^c	00±00 ^d	4995±06 ^a	6713±46 ^b
S+MG (1%)	25±0.1 ^a	3.8±0.2 ^{ba}	2747±33 ^b	2713±7 ^b	34±40 ^b	3652±715 ^b	6365±722 ^c
S+MG (1.5%)	24±0.1 ^a	3.9±0.6 ^{ab}	1831±57 ^c	1817±36 ^c	15±21 ^a	3022±650 ^c	4838±686 ^d
SC+MG (1.5%)	25±0.1 ^a	4.4±0.4 ^a	1755±20 ^c	1755±20 ^c	00±00 ^d	5039±401 ^a	6794±381 ^b

S-sorghum flour, SC-Sorghum coarse flour, control- durum semolina, MG-mono-glycerides.

* At process moisture 39.5%

** At process moisture 44.7%

*** At process moisture 47.9%

Table 4.10: Pasting properties of extruded pasta made from different flours formulated with salt, MG and corn starch.

Treatments	PTc (°C)	PTm (mins)	PV (cP)	TV (cP)	BD (cP)	SB (cP)	FV (cP)
Control	25±0.1 ^a	4.1±0.1 ^b	3847±60 ^a	3795±32 ^a	53±28 ^a	5468±725 ^b	9263±757 ^a
S+MG (1%)	25±0.1 ^a	3.8±0.1 ^c	2612±24 ^b	2612±24 ^b	00±00 ^c	3671±1152 ^c	6283±1176 ^b
T+MG (1%)	25±0.0 ^a	4.2±0.3 ^b	2213±110 ^{bc}	2189±105 ^c	24±06 ^b	3478±530 ^c	5667±426 ^c
T+CS (10%)+MG (1%)	25±0.0 ^a	3.9±0.3 ^{bc}	2544±65 ^b	2515±58 ^b	29±07 ^b	3859±355 ^c	6374±413 ^b
M+MG (1%)	25±0.2 ^a	4.9±0.0 ^a	1542±10 ^c	1152±10 ^d	00±00 ^c	5002±64 ^b	6154±074 ^c
M+CS (10%)+MG (1%)	25±0.2 ^a	4.5±0.0 ^{ab}	946±17 ^d	946±17 ^d	00±00	7874±456 ^a	8820±473 ^a

S-sorghum flour, SC-Sorghum coarse flour, control- durum semolina, T-teff flour, M-millet flour, MG-mono-glycerides, CS corn starch.

Table 4.11: Hardness of uncooked sorghum pasta, firmness and stickiness of values of cooked sorghum pasta.

Treatments	3PBT (Kg)	Firmness (kg)	Stickiness (Kg)
Control	3.00±0.80 ^a	5.58±1.82 ^a	0.58±0.04 ^a
S+MG (0.5%)*	1.38±0.22 ^d	4.55±1.67 ^b	0.21±0.02 ^{bc}
S+MG (0.5%)**	1.75±0.49 ^c	5.60±2.00 ^a	0.29±0.36 ^b
S+MG (0.5%)***	2.47±0.97 ^b	5.54±1.82 ^a	0.16±0.00 ^c
S+MG (1%)	1.16±0.28 ^e	4.47±1.73 ^b	0.12±0.00 ^c
S+MG (1.5%)	1.10±0.31 ^e	4.20±1.09 ^c	0.10±0.00 ^c
SC+MG (1.5%)	1.34±0.49 ^{de}	4.01±1.47 ^d	0.18±0.00 ^{bc}

S-sorghum flour, SC-Sorghum coarse flour, control- durum semolina, MG-mono-glycerides.

* At process moisture 39.5%

** At process moisture 44.7%

*** At process moisture 47.9%

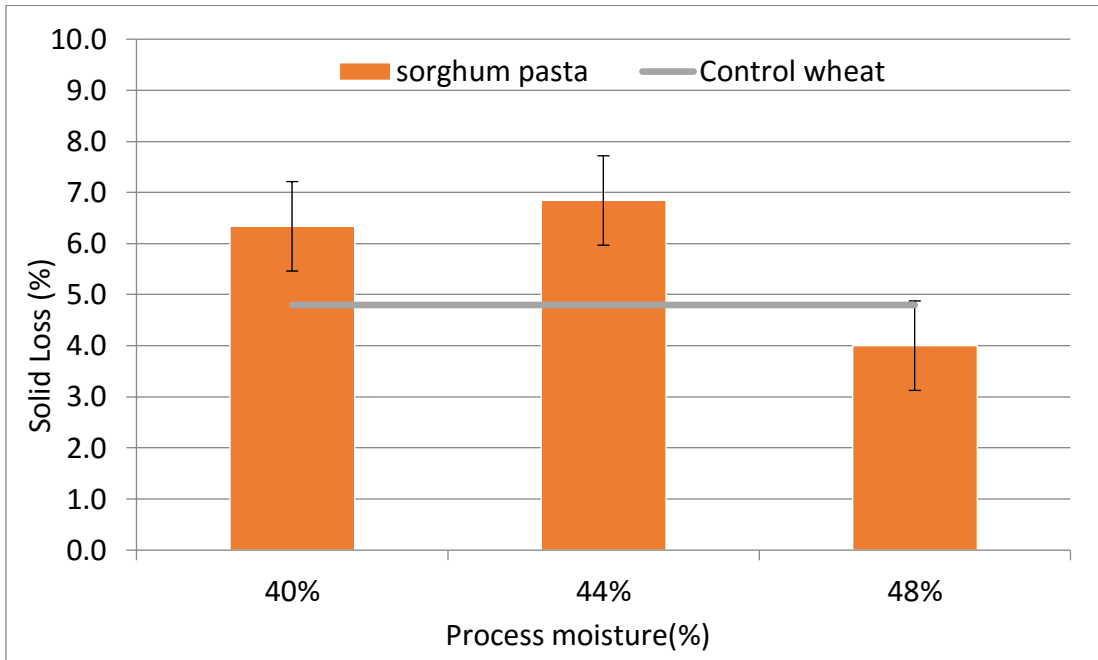
Table 4.12: Hardness of uncooked pasta, firmness and stickiness of values of cooked pasta formulated with sorghum, millet and teff flour.

Treatments	3PBT (Kg)	Firmness (kg)	Stickiness (Kg)
Control	3.35±0.77 ^a	7.53±1.51 ^a	0.22±0.02 ^c
S+MG (1%)	1.33±0.40 ^c	5.22±1.80 ^b	0.12±0.00 ^d
T+MG (1%)	1.60±0.48 ^b	3.20±1.09 ^d	0.37±0.03 ^a
T+CS (10%)+MG (1%)	1.66±0.67 ^b	3.87±1.37 ^c	0.30±0.01 ^{bc}
M+MG (1%)	0.73±0.19 ^d	4.61±1.45 ^{cb}	0.22±0.01 ^c
M+CS (10%)+MG (1%)	0.88±0.23 ^d	4.41±1.79 ^c	0.18±0.01 ^{cd}

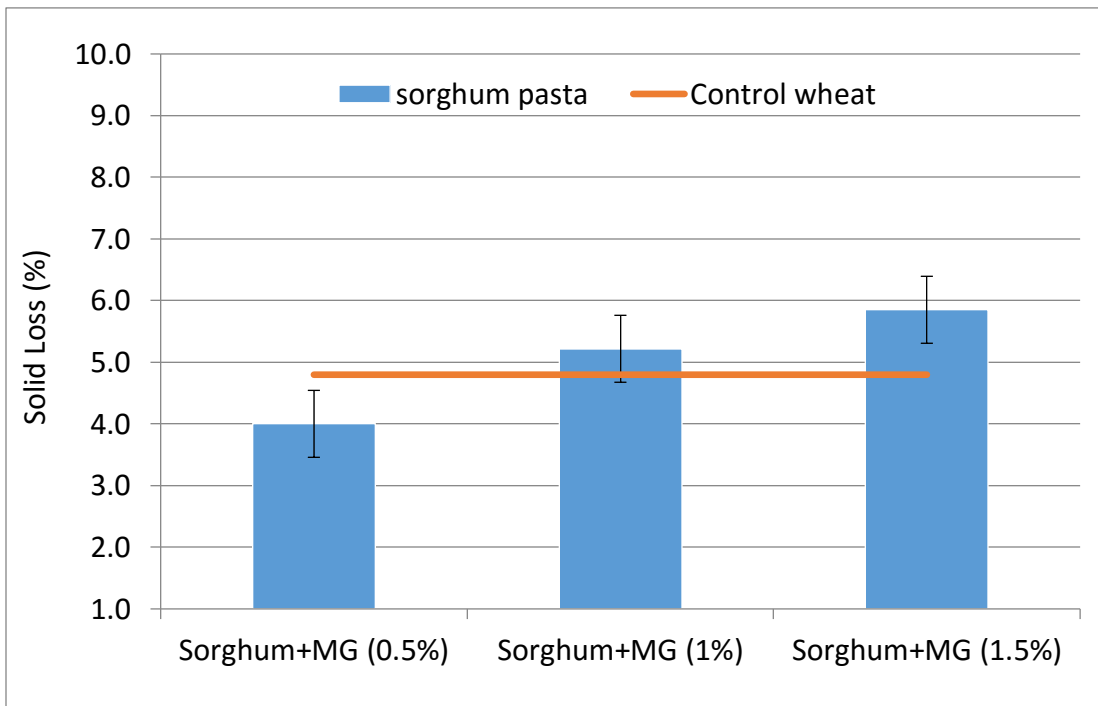
S-sorghum flour, SC-Sorghum coarse flour, control- durum semolina, T-teff flour, M-millet flour, MG-mono-glycerides, CS corn starch

Figure 4.1: Cooking loss comparison of different pasta processed at different process conditions and with different flours.

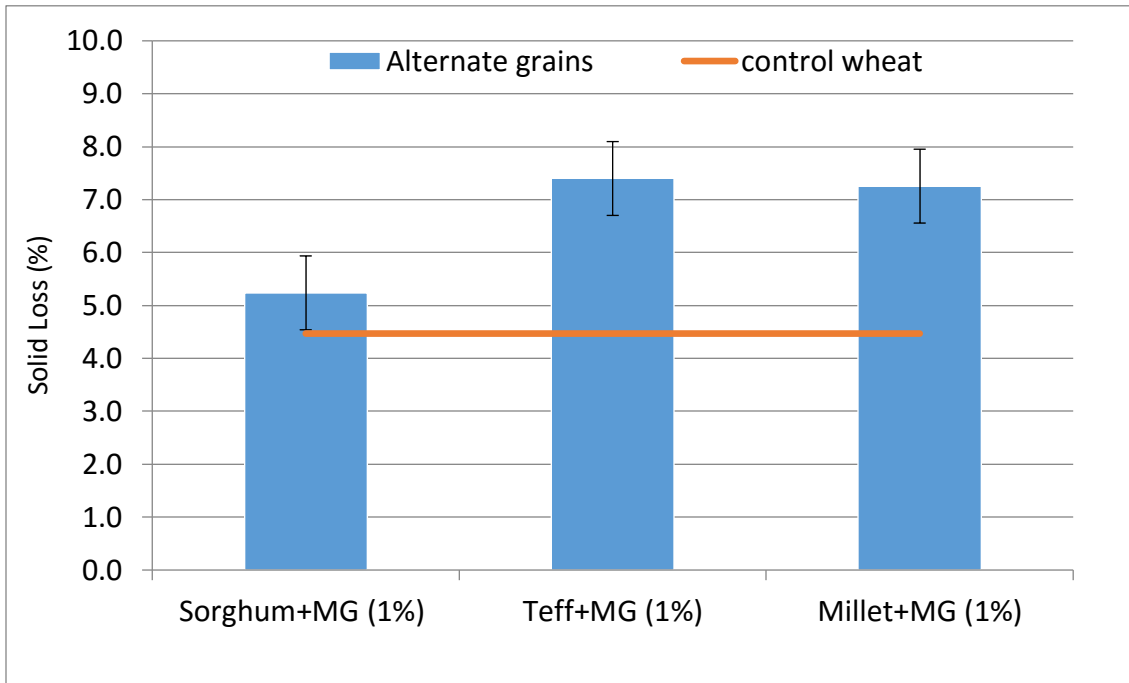
a) Average solid loss of sorghum pasta at different process moistures.



b) Average solid loss of sorghum pasta at different MG levels.



c) Average solid loss of pasta prepared from wheat, sorghum, teff and millet flour.



d) Average solid loss of teff and millet pasta formulated 10% native corn starch.

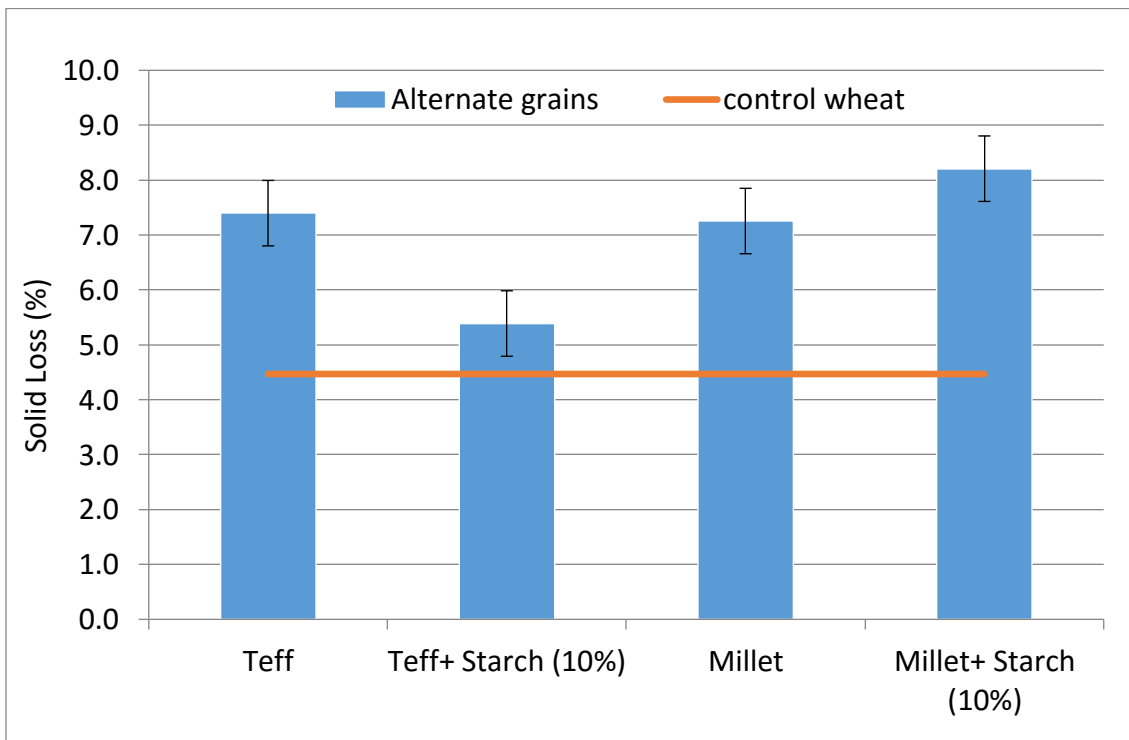
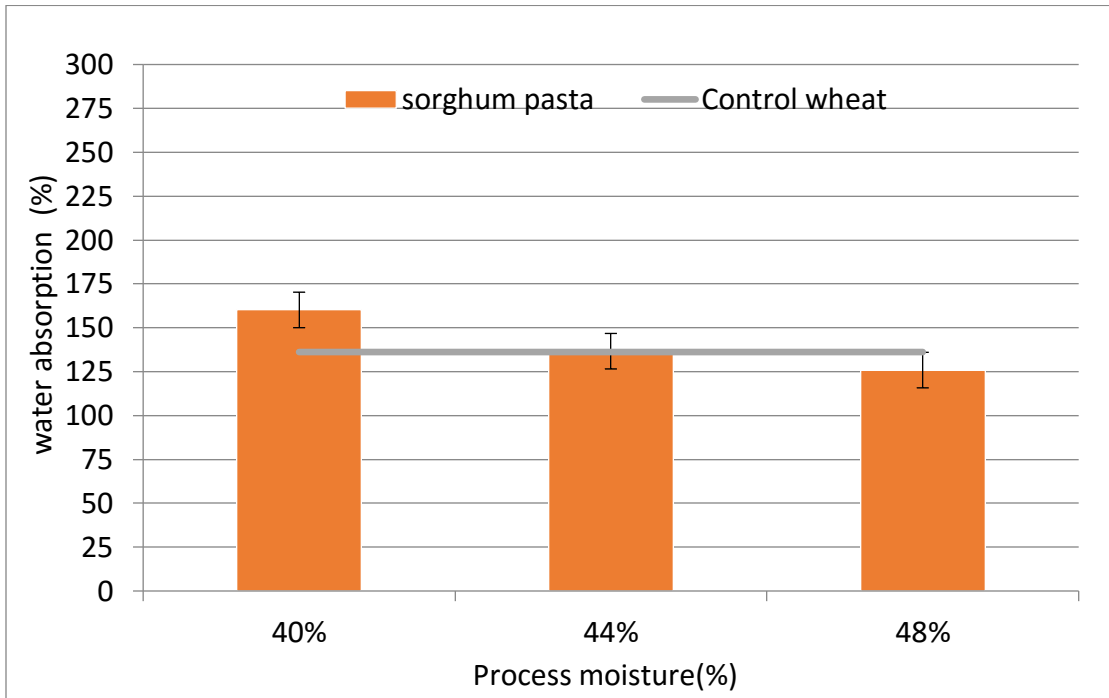
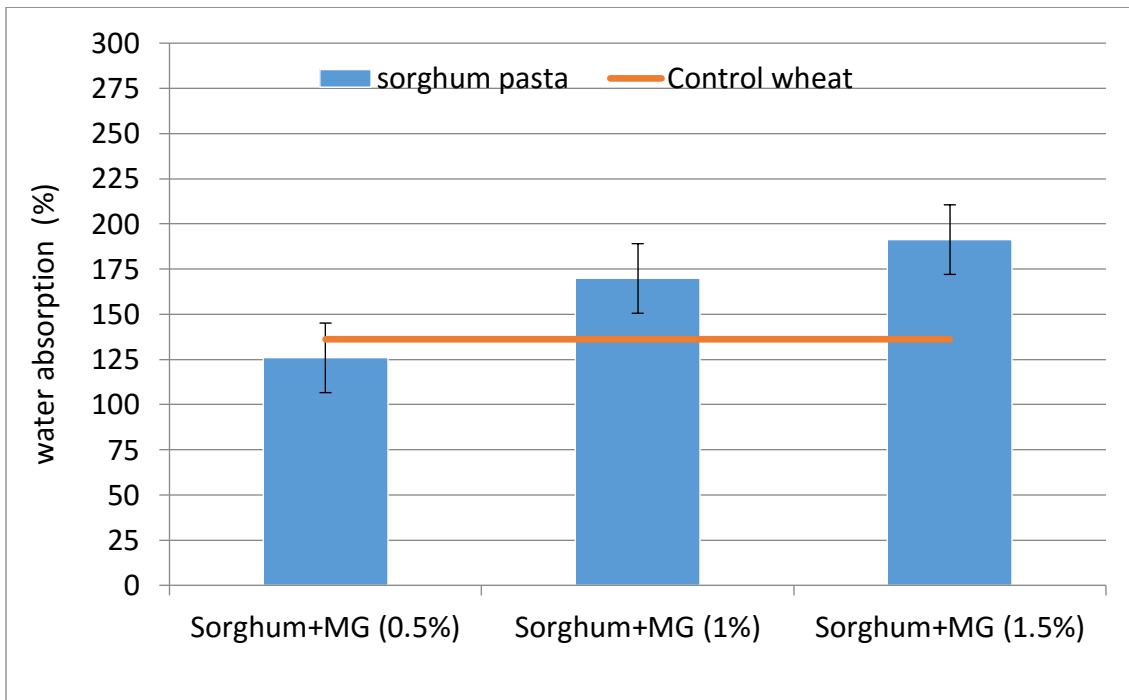


Figure 4.2: Water absorption comparison of different pasta processed at different process conditions and with different flours.

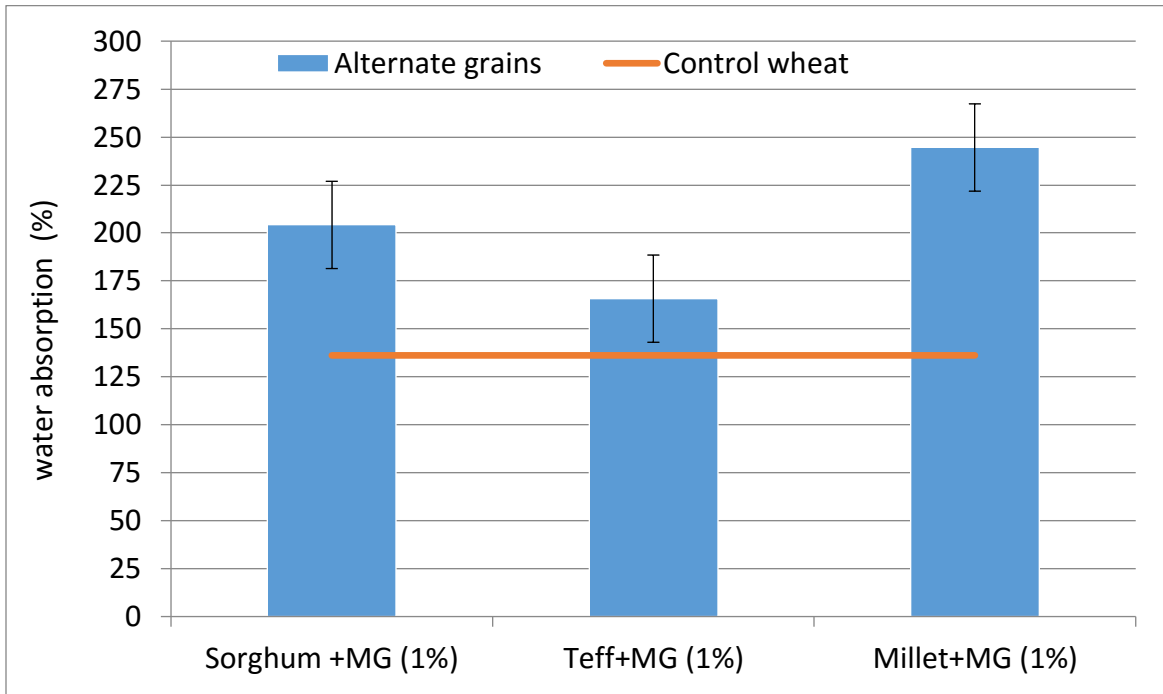
a) Average water absorption of sorghum pasta at different process moistures.



b) Average water absorption of sorghum pasta at different MG levels.



c) Average water absorption of pasta prepared from wheat, sorghum, teff and millet flour.



d) Average water absorption of teff and millet pasta formulated 10% native corn starch.

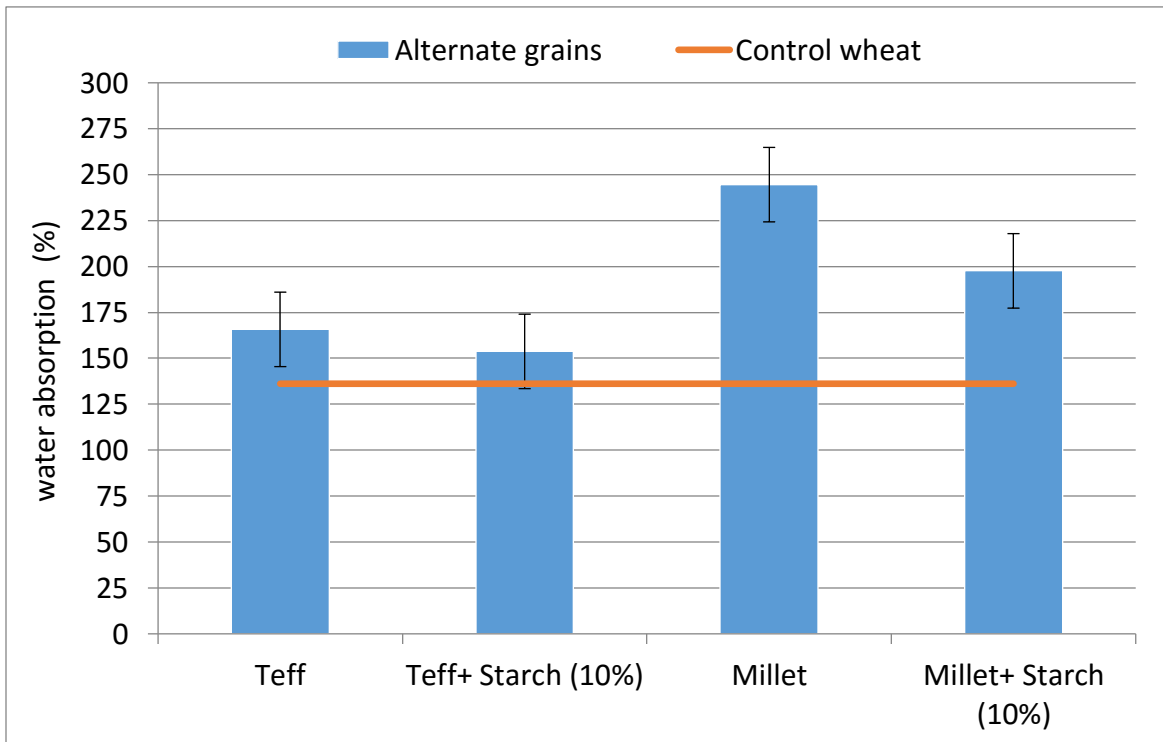
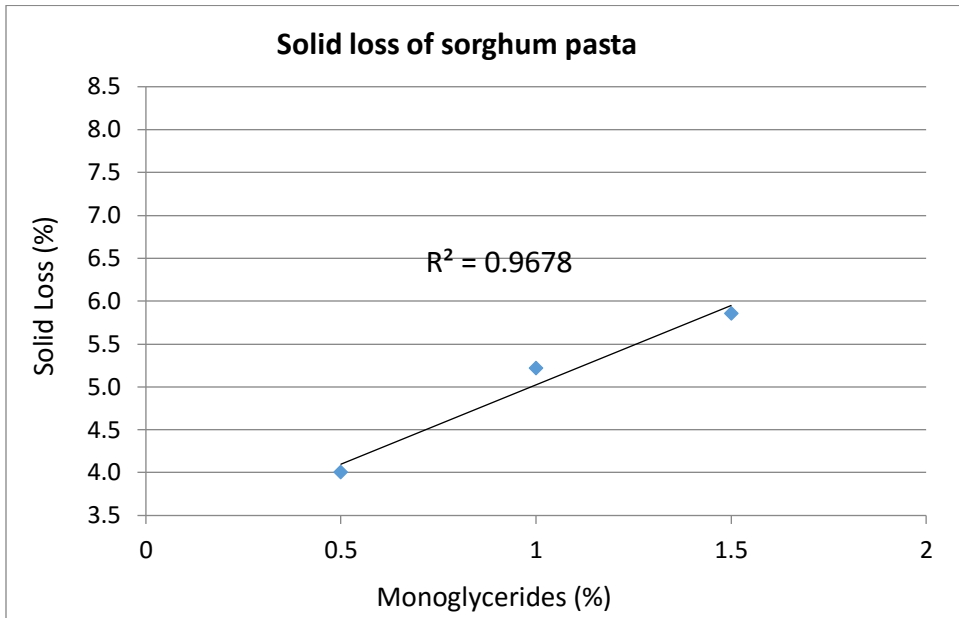
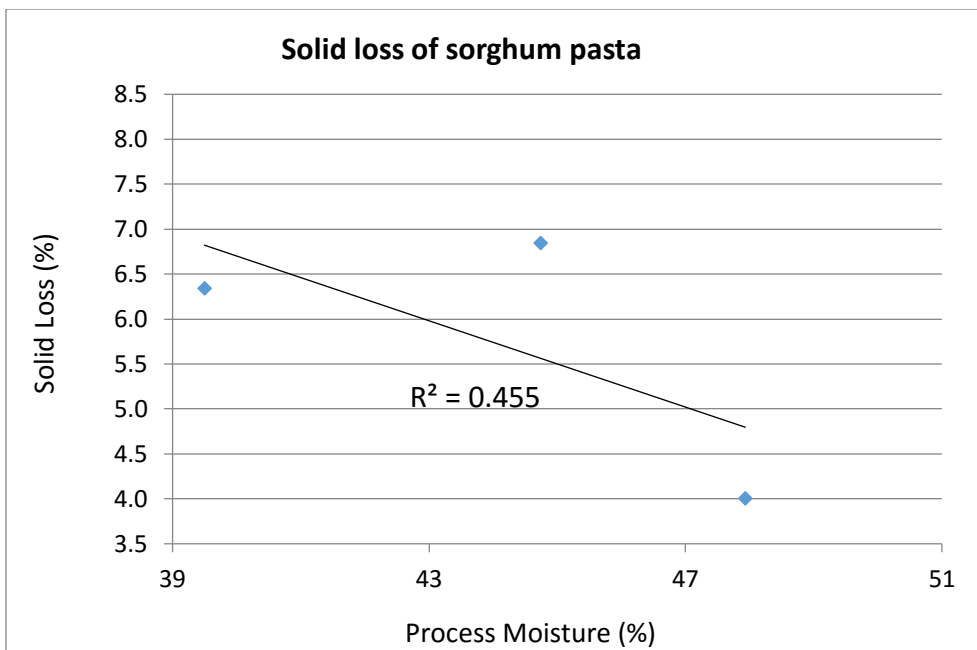


Figure 4.3: Correlations for solids loss for cooked pasta.

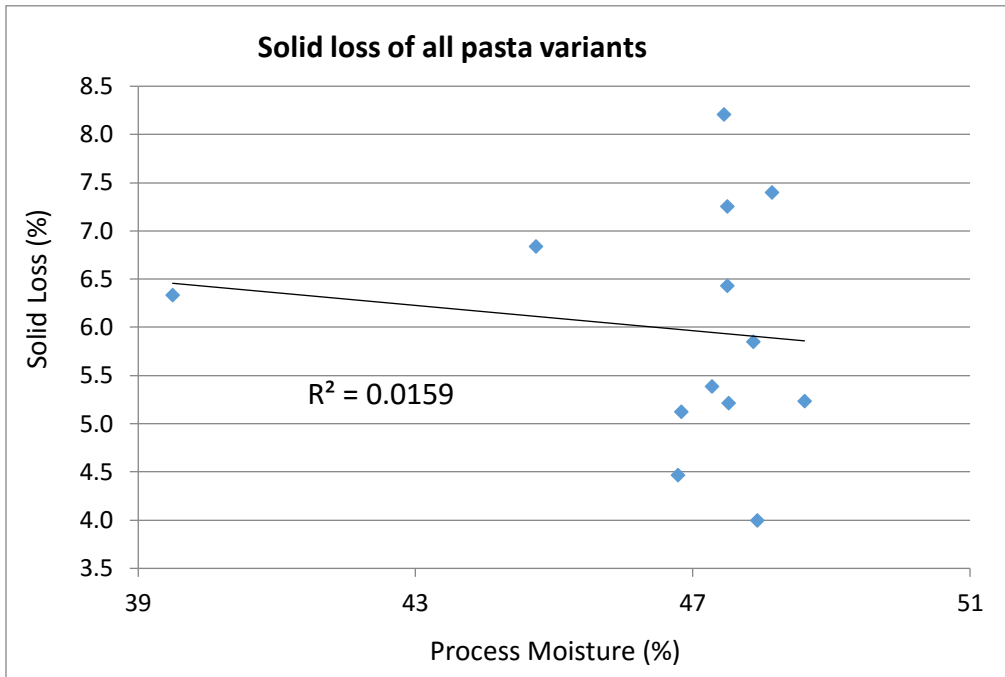
a) Correlation between solids loss (%) and mono-glycerides (%) for sorghum pasta.



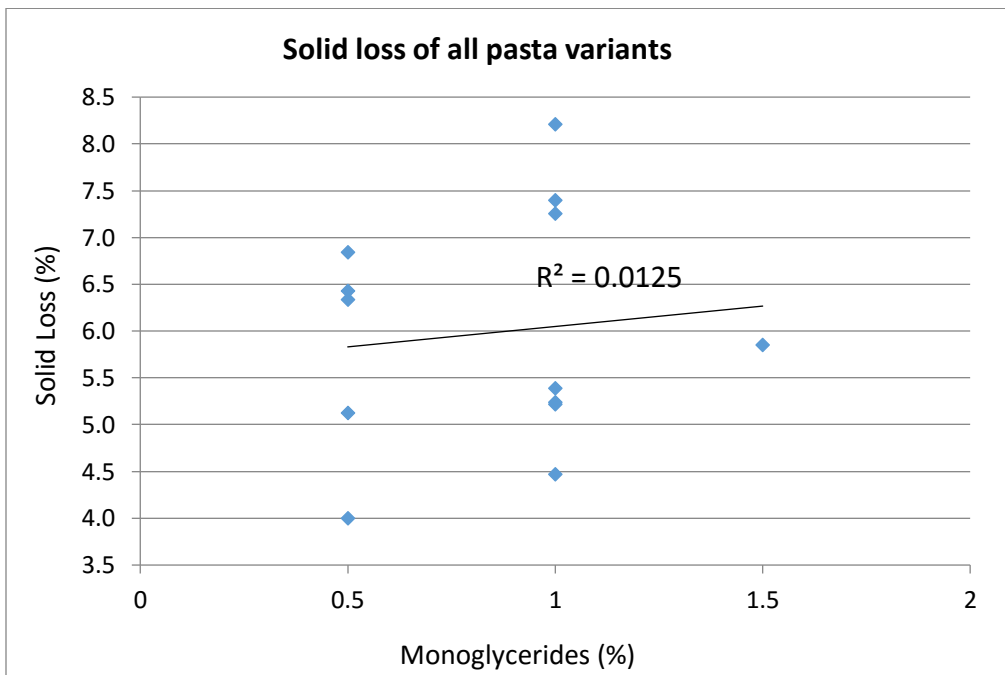
b) Correlation between solids loss (%) and process water (%) for sorghum pasta.



c) Correlation between solids loss (%) and process water (%) for all pasta variants.



d) Correlation between solids loss (%) and mono-glycerides (%) for all pasta variants.



e) Correlation between solids loss (%) and gelatinization (%) for all pasta variants.

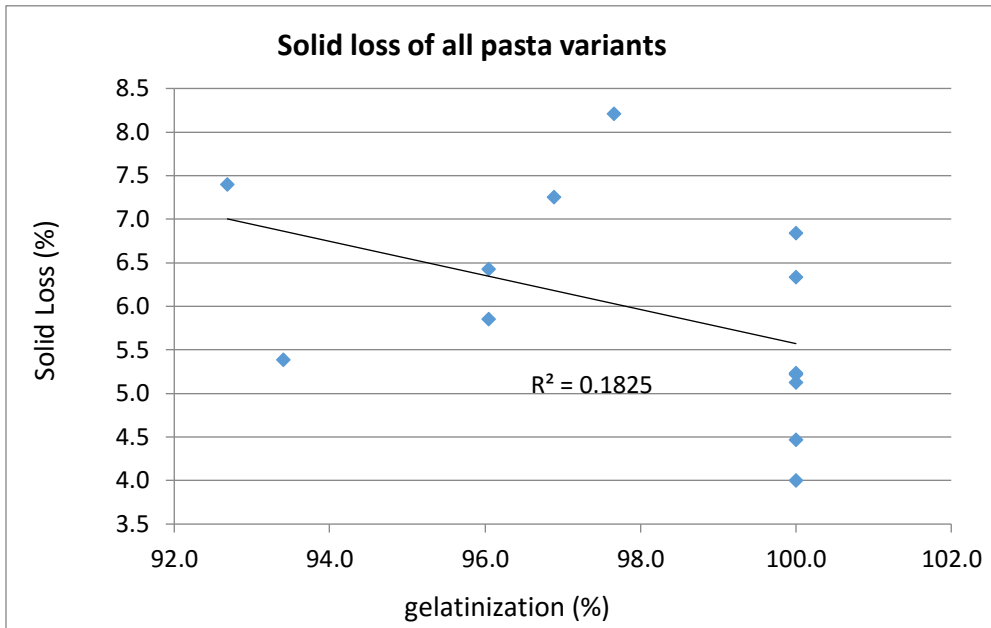
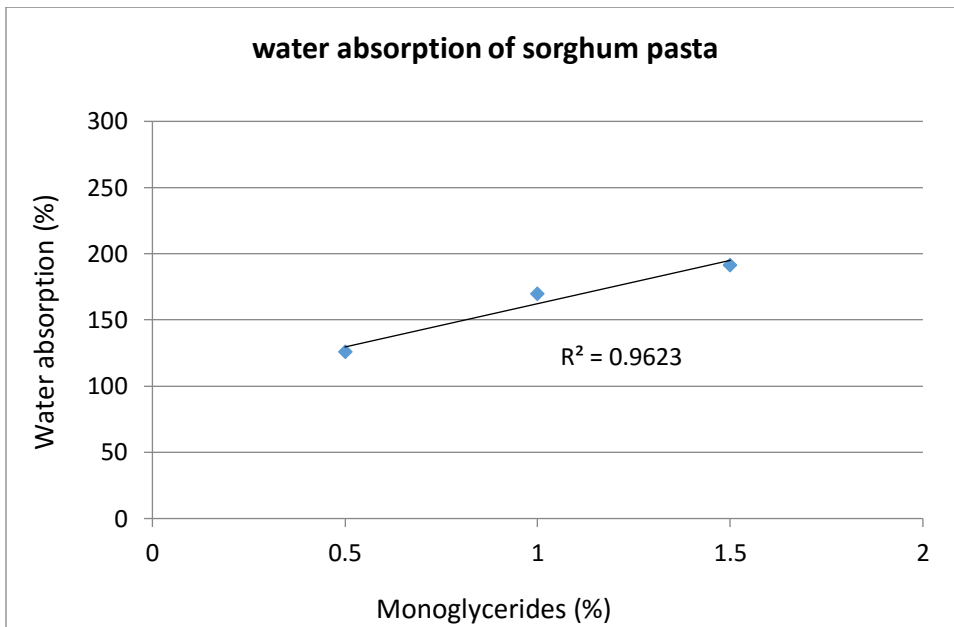
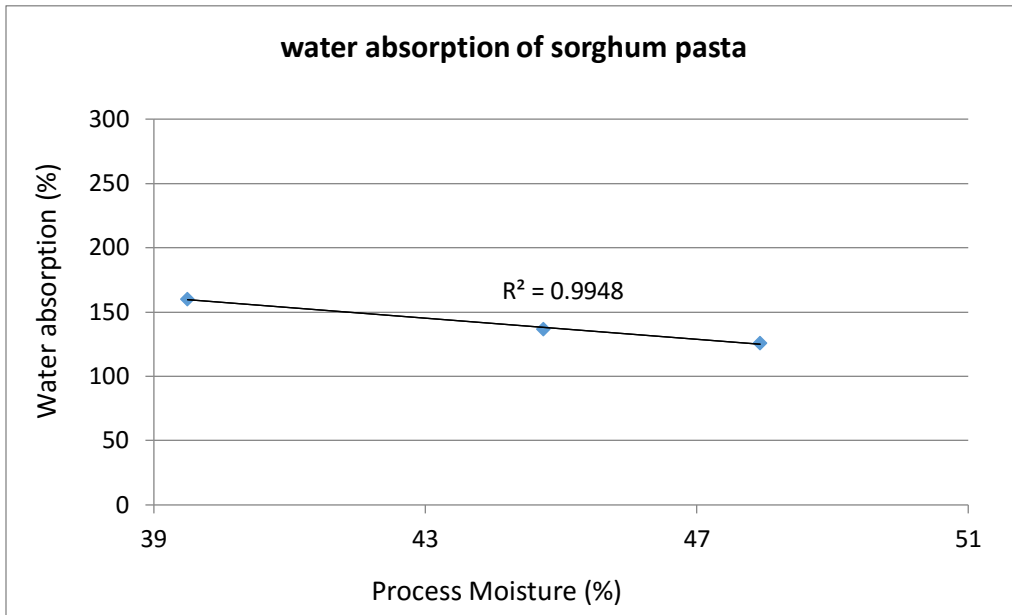


Figure 4.4: Correlations for water absorption for cooked pasta.

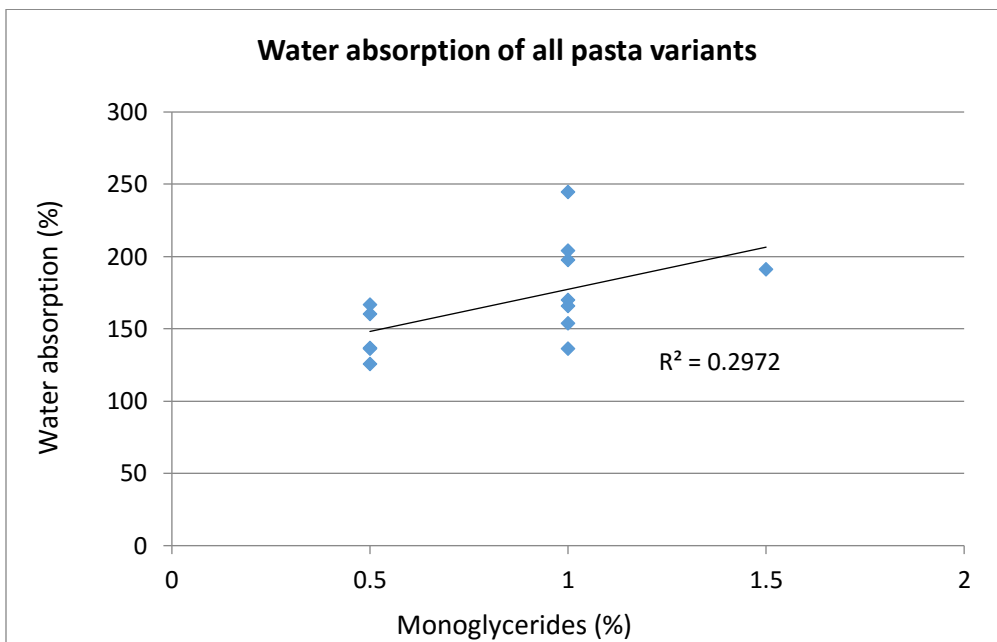
a) Correlation between water absorption (%) and mono-glycerides (%) for sorghum pasta.



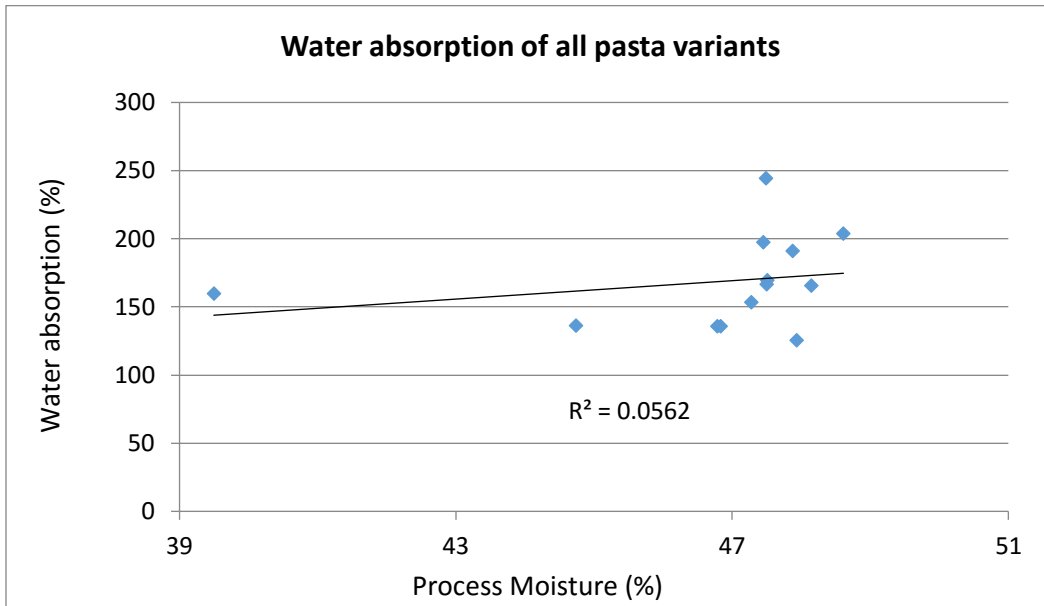
b) Correlation between water absorption (%) process water (%) for sorghum pasta.



c) Correlation between water absorption (%) and mono-glycerides (%) for all pasta variants.



d) Correlation between water absorption (%) and process water (%) for all pasta variants.



e) Correlation between water absorption (%) and gelatinization (%) for all pasta variants.

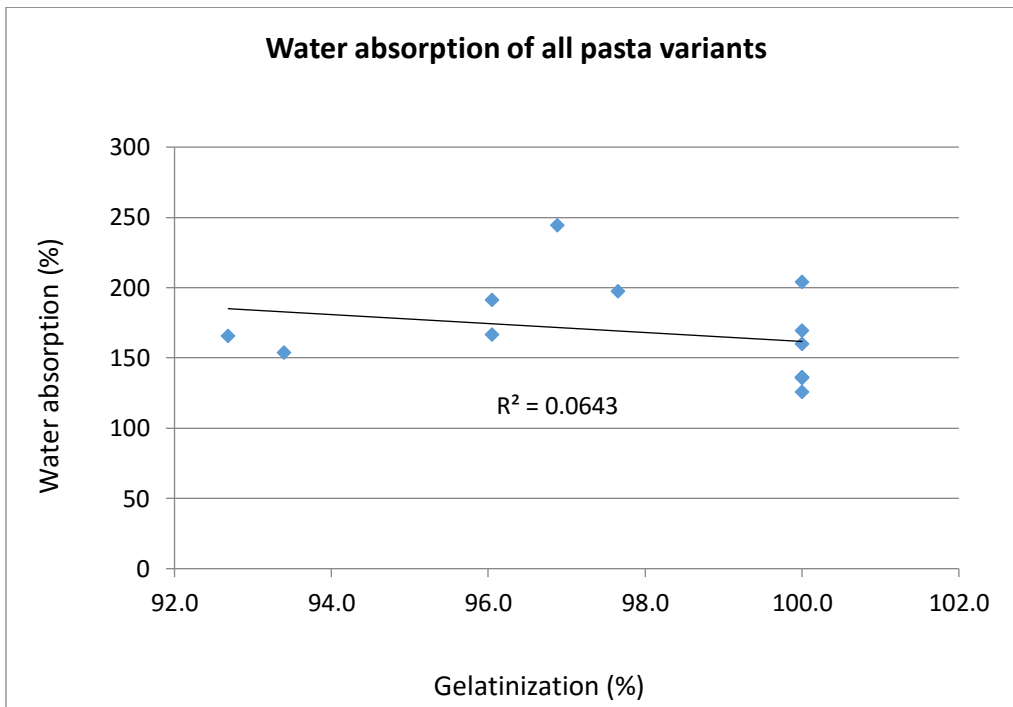
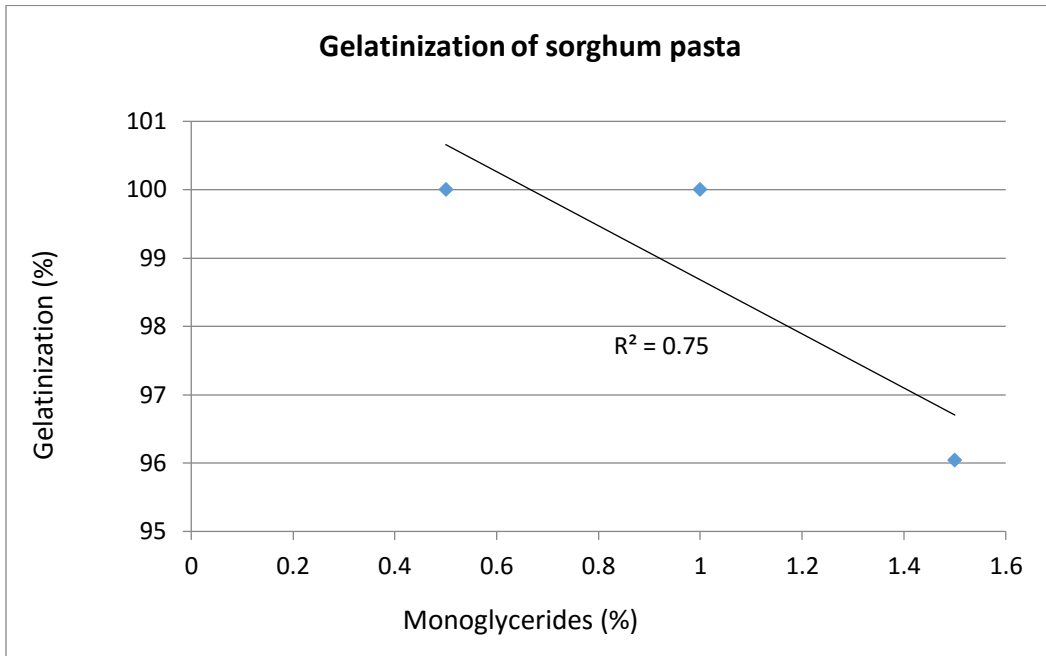
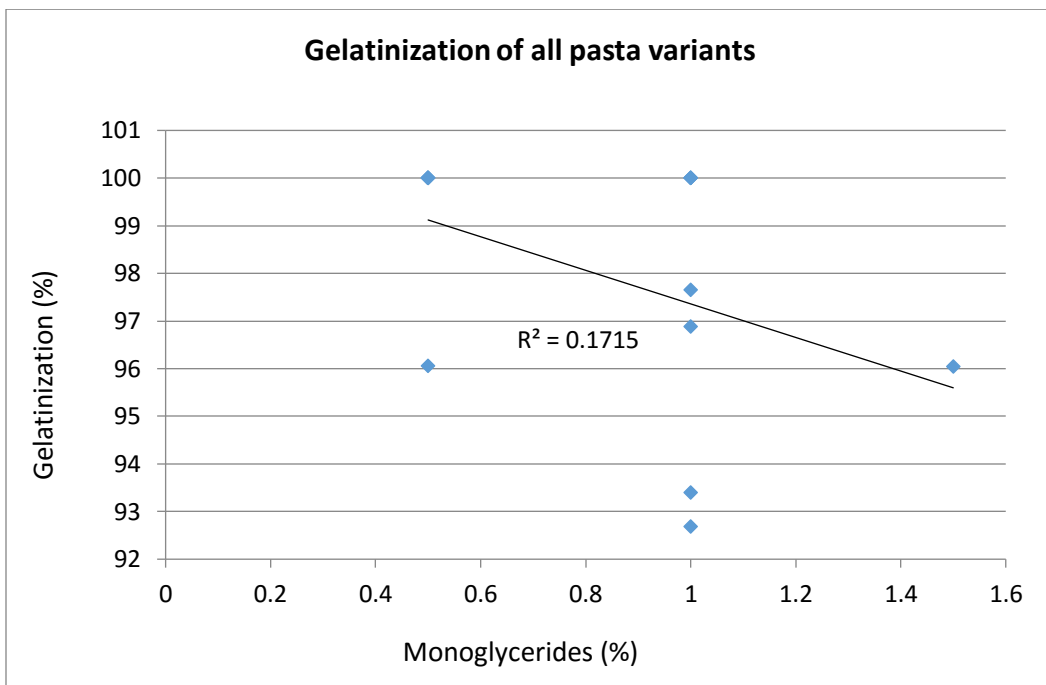


Figure 4.5: Correlations for gelatinization of precooked pasta.

a) Correlation between gelatinization and mon-glycerides (%) for sorghum pasta.



b) Correlation between gelatinization and mon-glycerides (%) for all pasta variants.



c) Correlation between gelatinization SME (KJ/KG) for sorghum pasta variants.

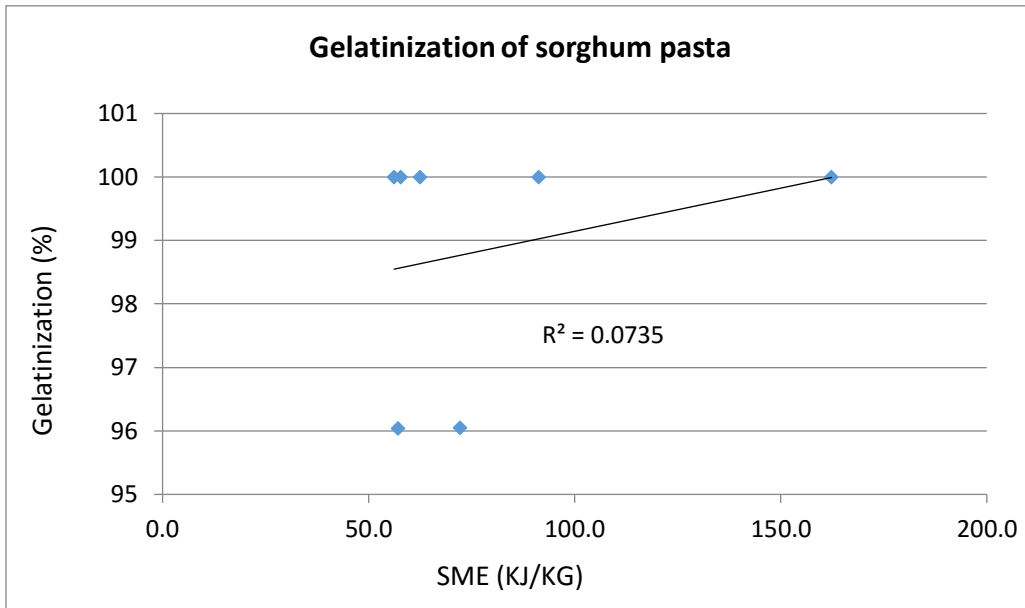
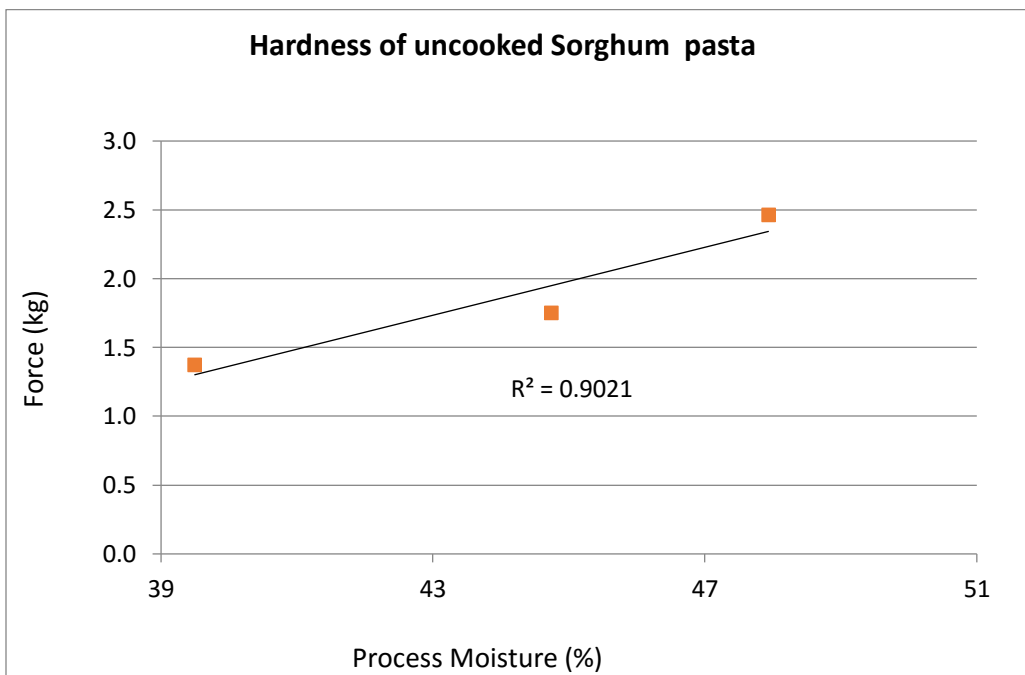
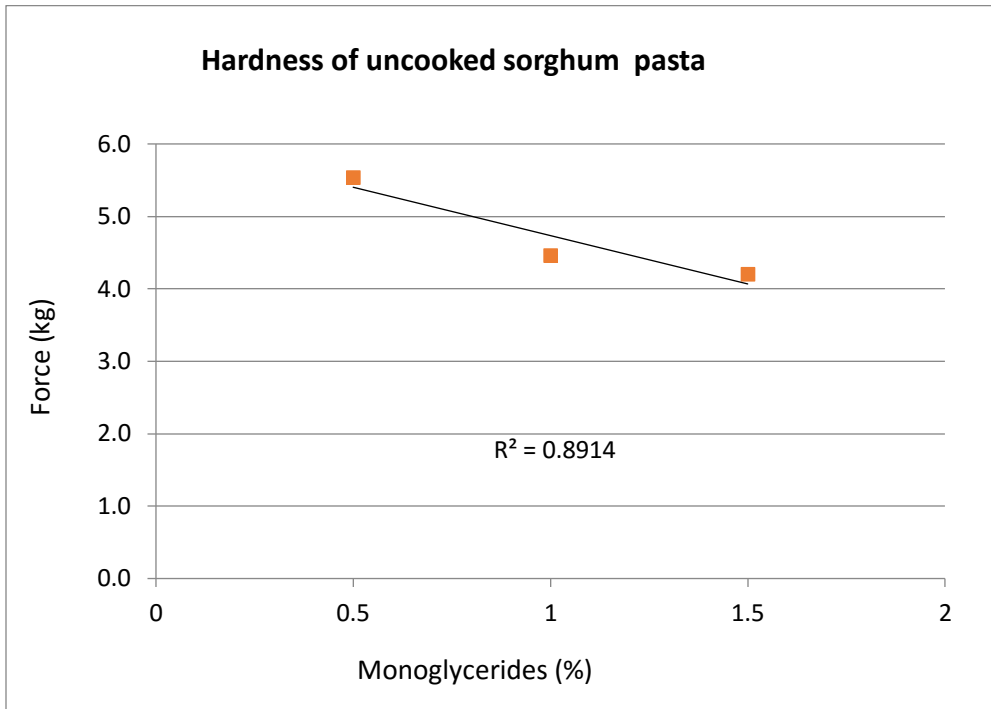


Figure 4.6:Correlations for hardness of uncooked pasta.

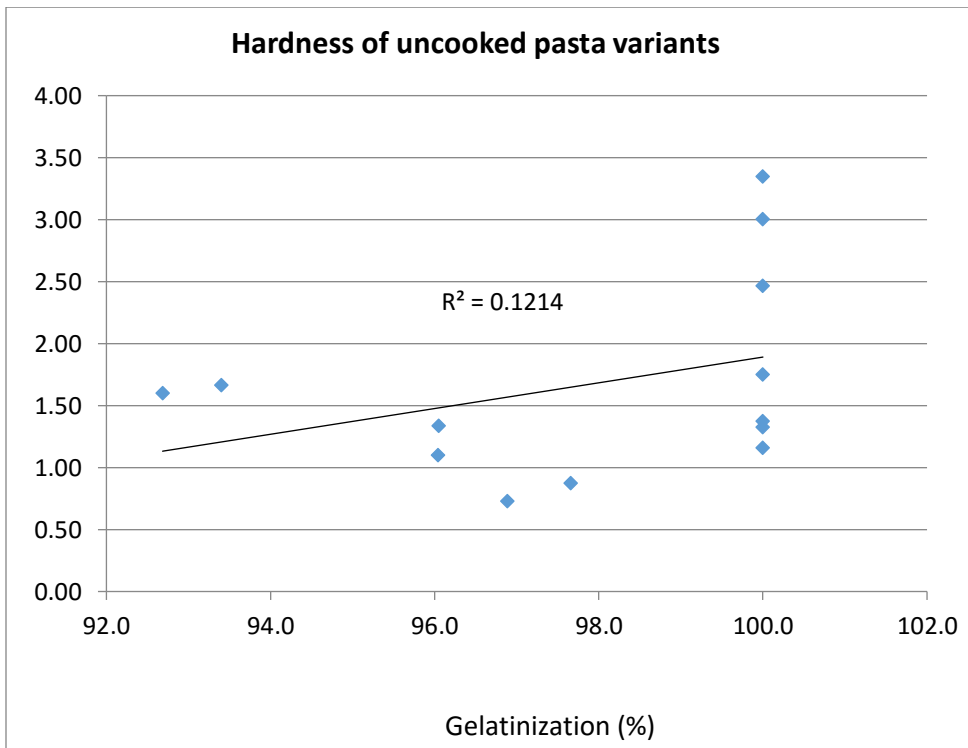
a) Correlation between hardness and process moisture (%) for sorghum pasta variants.



b) Correlation between hardness and mono-glycerides (%) for sorghum pasta variants.



c) Correlation between hardness and gelatinization (%) for all pasta variants.



d) Correlation between hardness and mono-glycerides (%) for all pasta variants.

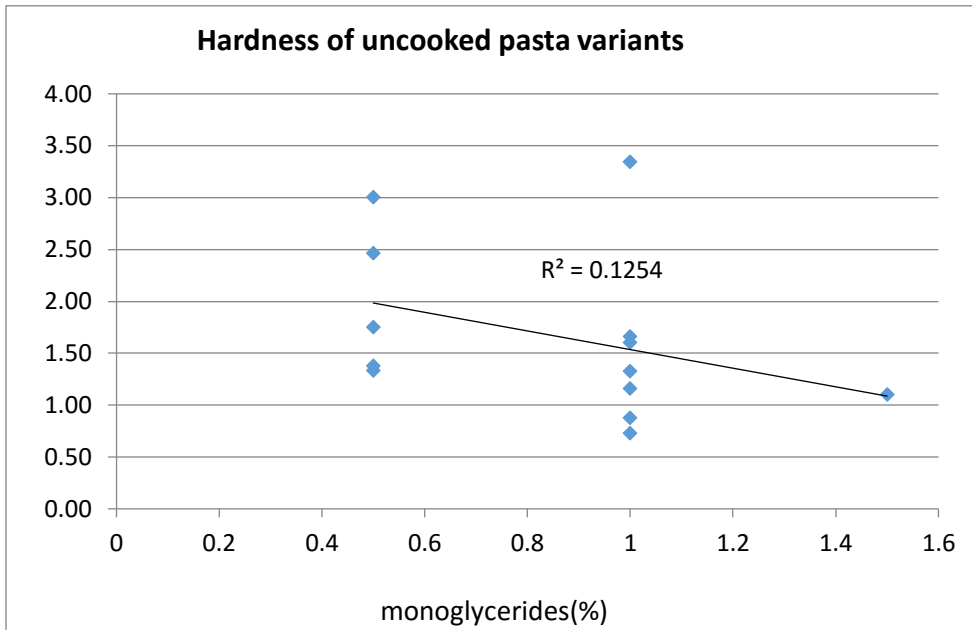
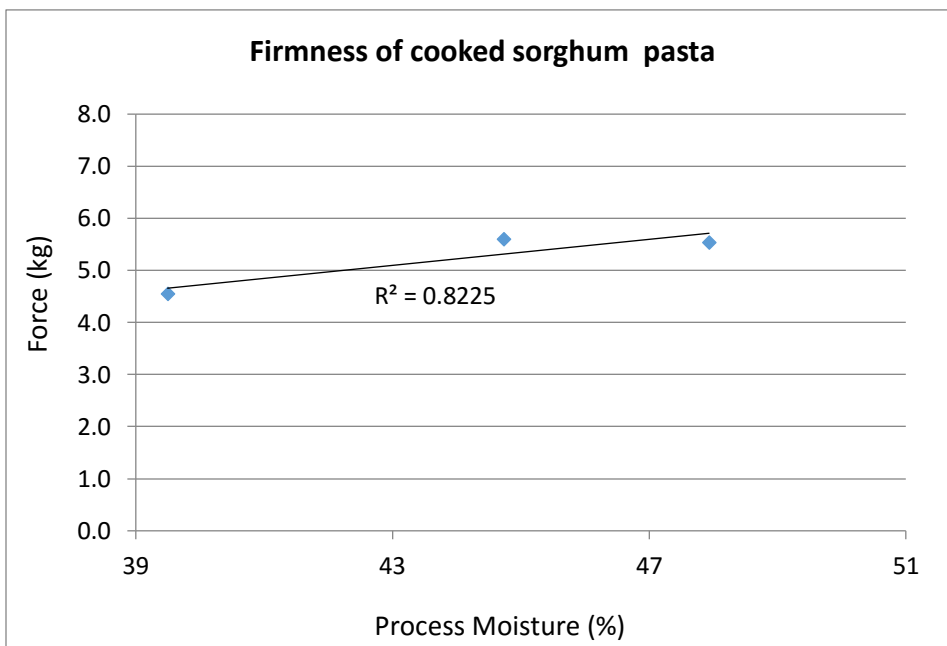
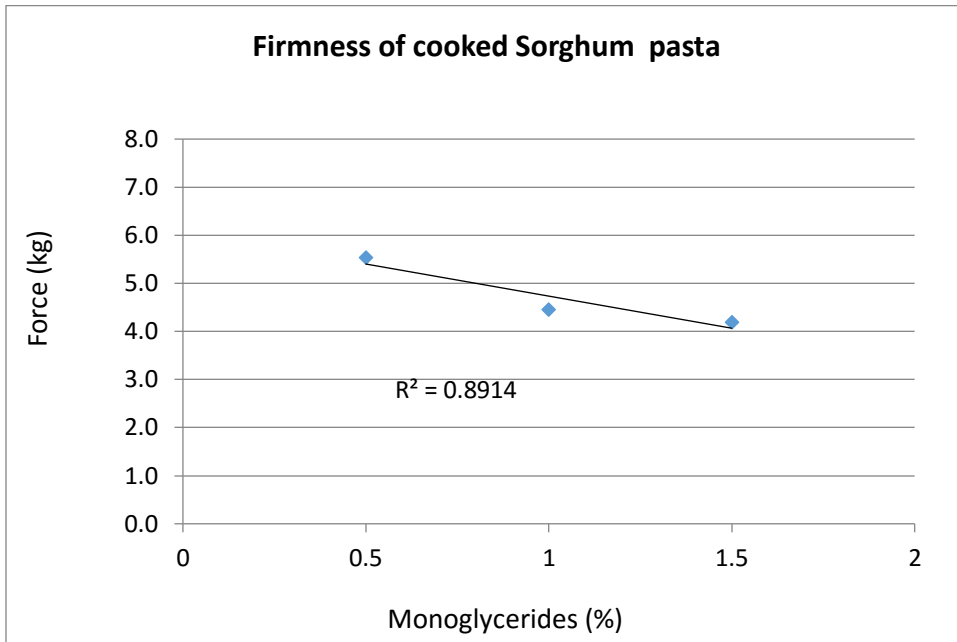


Figure 4.7:Correlations for firmness of cooked pasta.

a) Correlation between firmness and process moisture (%) for cooked sorghum pasta variants.



b) Correlation between firmness and mono-glycerides (%) for cooked sorghum pasta variants.



c) Correlation between firmness and gelatinization (%) for all cooked pasta variants.

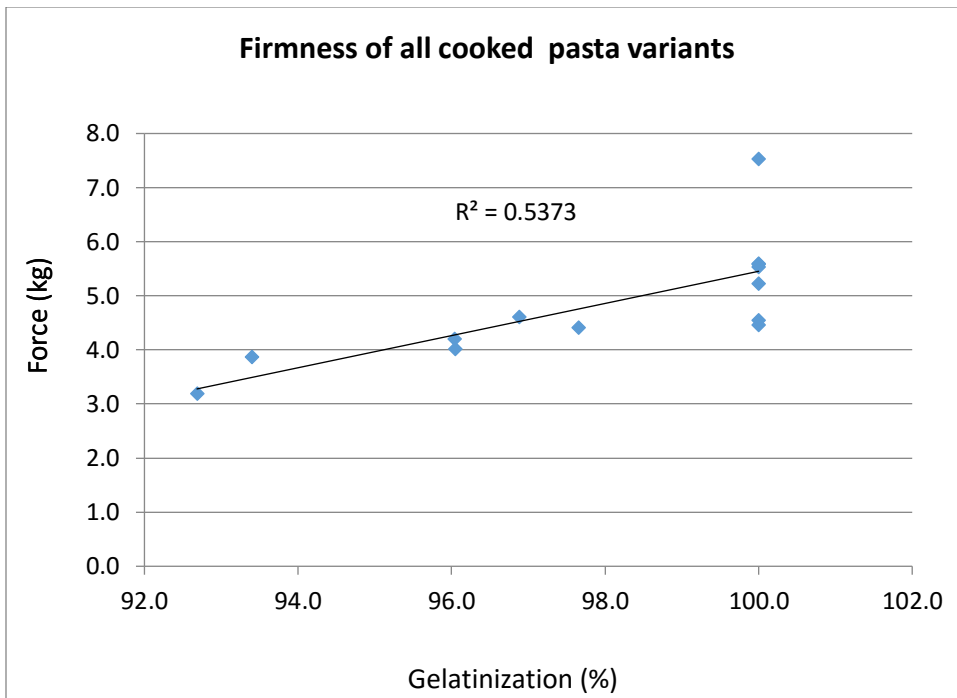
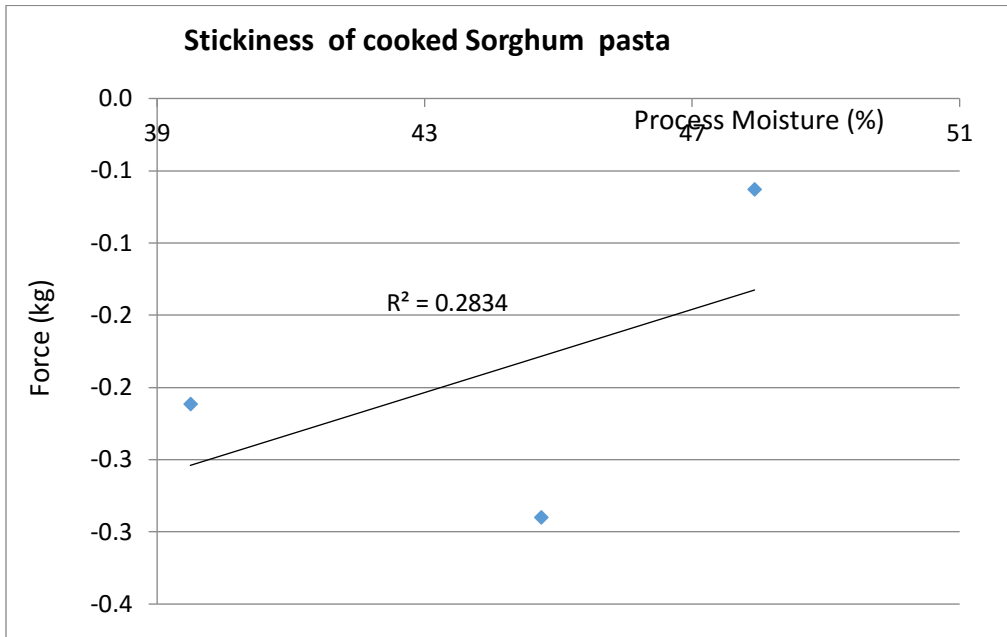
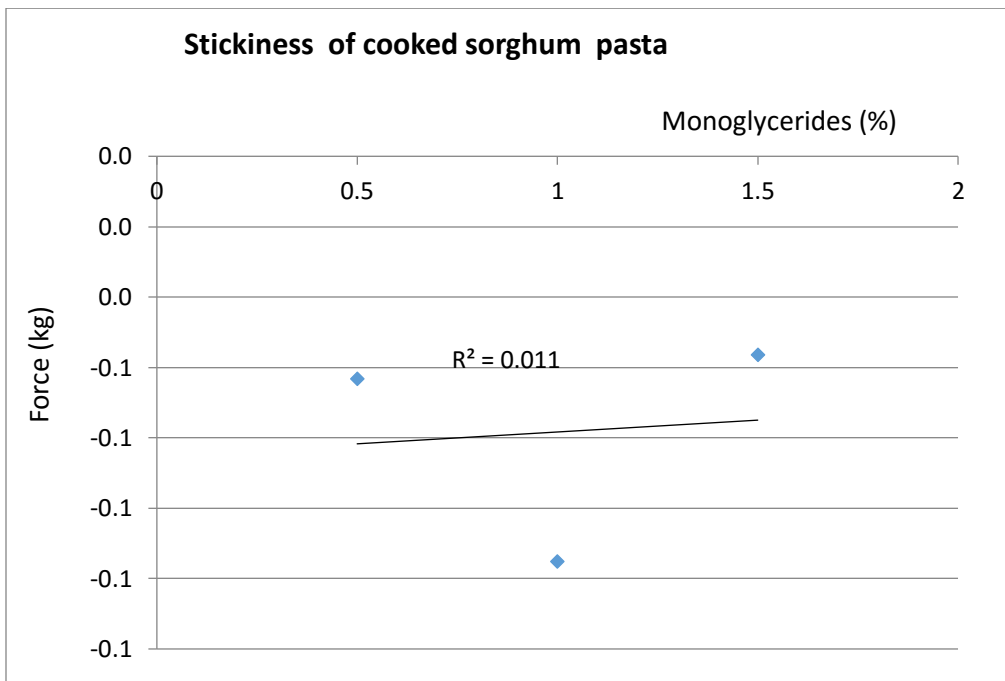


Figure 4.8: Correlations for stickiness of cooked pasta.

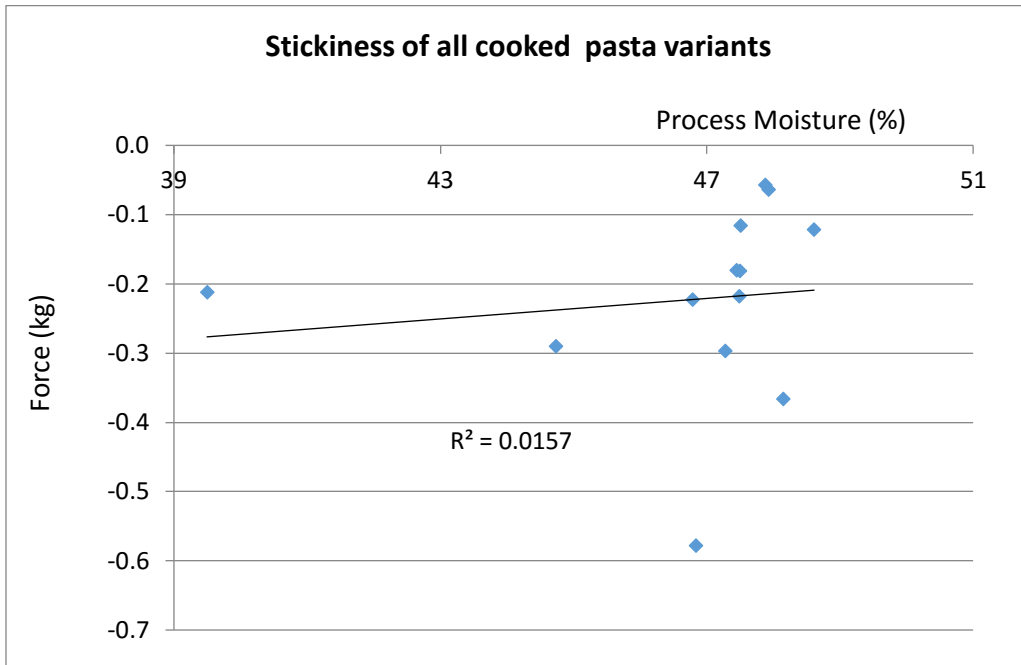
- a) Correlation between stickiness and process moisture (%) for cooked sorghum pasta variants.



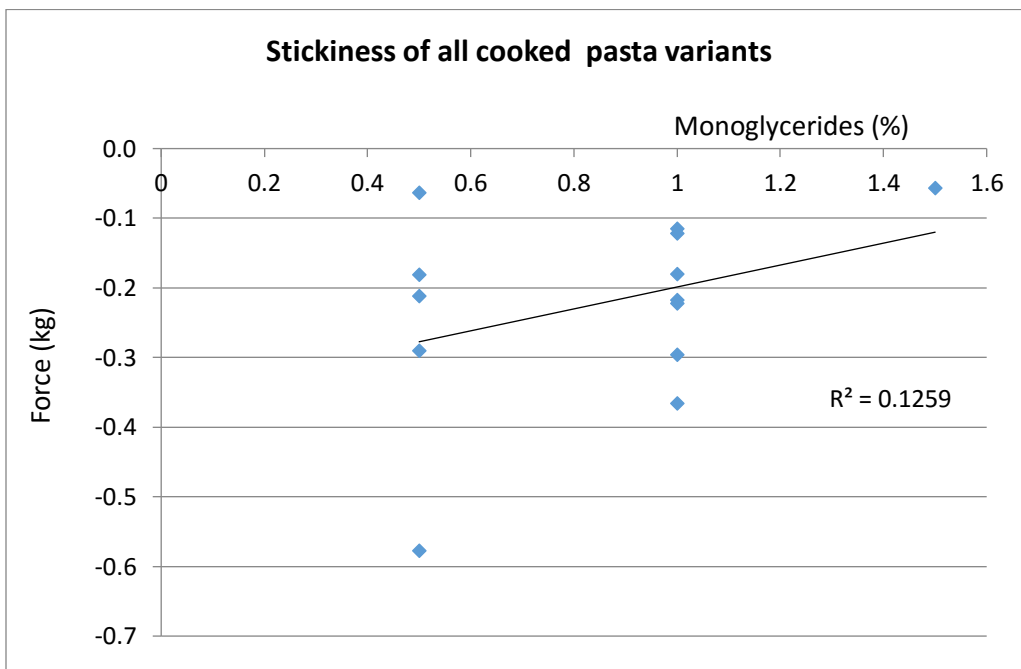
- b) Correlation between stickiness and mono-glycerides (%) for all cooked sorghum pasta variants.



c) Correlation between stickiness and process moisture (%) for all cooked pasta variants.



d) Correlation between stickiness and mono-glycerides (%) for all cooked pasta variants.



Chapter 5 - Resistant starch retention in expanded snack formulated with tannin sorghum and sumac bran in twin screw extrusion

Abstract

Different sorghum varieties and bran were extruded on pilot scale and lab scale to produce expanded extrudate. The effects of sorghum types and bran concentrations on extrudate expansion, specific mechanical energy (SME), piece density and retention of resistant starch (RS) were studied. The use of sumac sorghum and bran lowered process energy input, reduced expansion ratio, increased extrudate density and retained higher resistant starch over white sorghum flour. Increase in RS content retention for sumac flour and sumac bran extrudates were not significant ($p>0.05$), all the RS values were less than 1%. Extrudates processed at low shear and low energy input retained high RS in final products but RS values were not significantly higher ($p>0.05$). The use of sumac bran into sorghum flour significantly ($p<0.05$) lowered specific mechanical energy input (SME) into process. High fiber treatments were low in starch content which lowered viscosity lead to increase decrease in SME. Drop in SME can also be due to lubricating effect of high fat content of sumac bran. However, increase of bran percentage from 10% to 20% augmented SME but the increase was not significant ($p>0.05$).

Introduction

Sorghum (*Sorghum bicolor* (L.) Moench) is a significant food cereal in semi-arid tropics worldwide. The distinct advantage of sorghum is being drought-resistant and environment sustainability. With rising world population and declining water supplies, sorghum represents important crops for future. It is consumed as staple crop in many parts of the Africa and Asia (Murty and Kumar, 1995). Hence, sorghum acts as a principle source of energy, protein, vitamins and minerals for millions of the poorest people living in these regions (Klopfenstein and Hosney, 1995). The food uses of

sorghum are mostly traditions, and methods of processing may involve use of wet or dry heat (Joseph and Rooney, 2004). Traditionally, sorghum was used to prepare fermented and non-fermented porridges, flat breads and other alcoholic beverages. Sorghum grains are also popped and consumed as snacks or delicacies. In recent years, use of sorghum in human foods has surged because of its superiority in nutritional profile. The use of sorghum is extended to a variety of food products. It is milled into white flour to make expanded snacks, cookies breads and other ethnic foods.

Still sorghum remains as an underutilized crop in most developed countries, with being primarily used as animal feed (ICRISAT/FAO, 1996). Sorghum has considerable potential to be used in human food and beverages. In developing countries, commercial processing of these locally grown grains into value-added food and beverage products is an important driver for economic development (Taylor, 2004). In the developed countries, today there is a growing demand for gluten-free foods and beverages from people with coeliac disease and other intolerances to wheat gluten. Sorghum particularly could play an important role in area of food and beverage products.

Apart from being recommended as a safe food for coeliac patient's (Kasarda, 2001), sorghum contain substantial levels of a wide range of phenolic compounds. The health benefiting properties, especially antioxidant activity, and their use as nutraceuticals and functional foods are extraordinary (Dykes and Rooney, 2006). Tannins are such uniquely important phytochemical components of sorghum. They contain properties that have significant impacts on human health. Mostly tannins are associated with sorghum however 99% of sorghum varieties produced in United States are tannin free. Tannins protects sorghum plant from pest and diseases, tannin sorghum varieties are more tolerant to diseases than non-tannin varieties (Tipton et al., 1970; Rooney and Sullins, 1977 and Waniska et al., 1989). There is growing interest in sorghum natural antioxidants, polyflavans (tannins), and their potential health benefits. Special sorghum varieties containing high levels of condensed tannins (proanthocyanidins) are associated with various health benefits such as slow starch digestibility and antioxidants activity (Awika and Rooney, 2004; Awika et al., 2009). Hageman et al. (1998) and Tain

et al. (2012) reported that high molecular weight condensed tannins have more powerful antioxidant activity in vitro and in vivo over simple phenols and other natural antioxidants. Tannin concentration also reduces nutrient digestibility by interacting with proteins and digestive enzymes (Davis and Hosney, 1979).

Starch is the major component and main source of calories in cereal products. Amylose is the main starch component responsible in decreasing starch digestibility by forming resistant starch (Leeman et al., 2006). Decreasing starch digestibility is important phenomenon because it helps lower calorific intake, and generate health benefits against obesity and type 2-diabetes. Sorghum has shown lowest raw starch digestibility among cereals due to strong association between starch granules and endosperm proteins (kafirins). The presence of kafirin impedes α -amylase accessibility to starch (Rooney et al., 1986). The interaction between starch and cross-linked kafirins lowered cooked sorghum flour starch digestibility compared to corn (Zang et al., 1998).

Other components of sorghum such as polyphenols also decrease in vitro starch digestibility by inhibiting enzymes by interacting with starch (Hargrove et al., 2011; Thompson and Yoon, 1984). Lower molecular weight phenolic compounds including gallic acid, ferulic acid, and catechins were reported to change functional properties of starch by interacting with starch molecules (Wu et al., 2009; Beta and Corke, 2008). Barros et al. (2012) reported that interaction between proanthocyanidins (condensed tannins) and starch increased resistant starch and decreased in vitro digestibility. They demonstrated that sorghum tannins interacted with amylose component of starch and, decreasing enzymatic starch digestibility. Mkandawire et al. (2013) presented that high-molecular-weight proanthocyanidins reduced α -amylase activity more significantly than lower-molecular-weight proanthocyanidins. This study is aimed to evaluate effects of different extrusion processing conditions on resistant starch retention in sorghum based snacks formulated with different concentrations of tannins rich sumac sorghum and sumac bran.

Materials and Methods

Raw Materials

Coarse white pearled decorticated sorghum flour with average particle size of 125 μ m of lot number KSU-17042709, sumac sorghum bran lot number KSU-170613-24 and sumac whole grain sorghum flour lot number KSU-170613-24 were purchased from Nulife Market, Scott city, Kansas. Distilled mono-glycerides DIMODAN HS K-A was obtained from Danisco USA Inc., New Century, Kansas USA Lot number 1142945586, product no 810773. Dry ingredients were premixed in a Hobart planetary mixer and water was added simultaneously to achieve target feed moisture. Total mixing time was 5 mins for all treatments. All mixed hydrated ingredients were transferred into sealed plastic zipper and were let to hydrated overnight in a refrigerator to achieve uniform water hydration. The tannins content of whole sumac flour was 21.97 \pm 0.45 and in sumac bran was 58.44 \pm 10.27 (Awika et al., 2003). The white sorghum coarse flour was tannin free.

Experimental Design

Lab Extrusion

Various blends of white sorghum flour, sumac flour and sumac bran (20%) were processed at three different moisture contents (20%, 28% and 36%). Expanded snack was produced using a lab scale co-rotating twin screw (American Leistritz, Somerville, NJ) with L/D ratio of 30:1, screw diameter of 18 mm equipped with a die diameter of holes 4 mm. Raw materials mix were metered into extruder with a twin screw volumetric feeder (K-Tron, Model K2VT20, North America, Pitman, NJ, USA). The feed rate was calibrated for each treatment independently and was kept at 3 kg/h. The screw speed was kept constant at 500 RPM and barrel temperatures were between 50- 100 degree Celsius. Once out from die the product was cut into long strands (25 cm) with the stainless-steel kitchen knife and packed into zipper pack plastic bags. The product was immediately stored in freezer to prevent starch retro-gradation. Drying was carried out

at 70°C for 40 mins in hot air oven. Net motor load (torque) was used to represent the energy input going into the process.

Pilot Extrusion

Puffed expanded pillow shaped shells were produced in an industrial twin screw extruder X-52 (Wenger Manufacturing Inc., Sabetha, KS, USA) equipped with a differential diameter cylinder preconditioner (DDC2, Wenger Manufacturing Inc., Sabetha, KS, USA). A five-independent zoned 1326 mm long barrel fitted with 52 mm screw diameter screws with an L/D ratio of 25.5:1 was equipped in extruder assembly. A loss in weight single screw feeder (Wenger Manufacturing Inc., Sabetha, KS, USA) was adjusted to feed 60 kg/h into the preconditioner. The barrel temperatures were in the range of 33 to 78°C. The heating of jacketed barrel was controlled by oil and heating elements. Water was added into the preconditioner, and the amount of water added into the preconditioner was ranged between 2.3 to 2.7 kg/h. The water flow rate into the preconditioner was controlled by remote flow transmitter, model RFT97121PNUR, RFT9712 S/N 59140 and mass flow sensor model-DS012H205SUP, sensor S/N 179066, meter type-21, make Micro Motion Inc., CO, USA. The preconditioner discharge temperature was in the range of 25 to 28°C. The screw speed of differential diameter cylinder preconditioner was kept constant at 297 RPM.

The process moisture content of the feed mix was adjusted in between 18.9% to 21.5% based on initial moisture content of blends and additional water injected into extruder barrel to achieve target moisture levels. The extruder water flow rate was also controlled by remote flow transmitter model-RFT97121PRU, RFT9712 S/N 70307, SEN S/N 203177 and mass flow sensor model-DS012S100SU, meter type-1, Micro Motion Inc., CO, USA. No steam was added into preconditioner and extruder during whole experiment. The screw speed of extruder was kept constant at 397 RPM. Thermocouples mounted at each zone of the extruder barrel were used to record respective (set and actual) zone temperatures from the control panel. The die temperature and pressure was also recorded directly using thermocouple and pressure

gauge respectively. The die temperature was in the range of 167 to 191°C and die head pressure ranged from 650 to 1100 per square inch gauge (psig). A cylindrical shaped die of diameter 8.0 mm was fitted on the die head. The expanded shell was sealed and crimped with the help of crimper cutter.

All the cut extrudates pieces were pneumatically conveyed to Wenger Double Pass Dryer/Cooler (Series 4800, Wenger Manufacturing Inc., Sabetha, KS, USA) operating at 76.7°C. The total retention time in the dryer was 30 minutes (8 minutes for top and 14 minutes for bottom belts). Cooling was accomplished by room temperature air with 8 minutes' retention time on cooling belt.

The specific mechanical energy (SME) for each treatment was calculated as follows:

$$SME = \frac{\frac{(\tau - \tau_0)}{100} \times P_{rated} \times \frac{N}{N_{rated}}}{\dot{m}}$$

Where, τ = operating torque (%); τ_0 = no-load torque (%); P_{rated} = rated power (37.3 kW), N = screw

speed (rpm); N_{rated} = rated screw speed (336 rpm), and \dot{m} = net mass flow rate of pasta at die exit (kg/s).

Extrudate Macrostructure

For every treatment, length (l_e), diameter (d_e) and mass (m_e) of 20 extrudates were measured and used to obtain radial expansion ratio (ER), specific length (l_{sp}) and piece density (PD), as described below.

where, Expansion ratio (ER),

$$ER = \frac{d_e^2}{d_d^2}$$

where, d_d = die diameter

Specific length (l_{sp}),

$$l_{sp} \left(\frac{m}{kg} \right) = \frac{l_e}{m_e}$$

Piece density (PD),

$$PD \left(\frac{kg}{m^3} \right) = \frac{4m_e}{\pi d_e l_e}$$

Determination of Resistant Starch

Quantification of the RS content was performed according to method certificated by AACC (2001) and AOAC (2000) using RS assay kit supplied by Megazyme International Ireland Ltd (Wicklow, Ireland). Briefly, enzymatic hydrolysis of non-resistant starch (NRS) was performed through the simultaneous action of pancreatic α -amylase (10 mg/mL) and amyloglucosidase (3 U/mL) by incubating sample for 16 h at 37°C. Subsequently, the NRS was separated by centrifugation, and pellet containing RS was purified with ethanol and solubilized with 2 mol/L KOH. The concentration of RS was measured at 510 nm, and the content was expressed as g/100 g sorghum flour on a dry weight basis. The results were obtained in analytical duplicate and are presented as mean \pm standard deviation.

Proximate Analysis of Raw Materials

The proximate composition of raw ingredients was determined using standard methods. This included determination of moisture (135°C for 2h; AACC 44-19), crude protein (based on nitrogen by combustion, 6.25X; AOAC 920.176), crude fat (petroleum ether extract method; AOCS Ba 3-38), ash (600oC for 2h; AOAC 942.05), crude fiber (AOAC 962.09); and total starch (glucoamylase method; AOAC 979.10). Starch, protein, fat,

ash and crude fiber contents were reported on dry basis percentage (% db) from replicates. Total carbohydrate was calculated by the difference method (Merrill and Watt, 1973).

Statistical Analysis

All the results were analyzed using analysis of variance (ANOVA) with general linear model procedure (SAS version 9.1, SAS Institute, Cary, North Carolina, USA). When significant effects ($p \leq 0.05$) were indicated by ANOVA, Tukey pairwise comparisons were conducted to distinguish which treatments differed significantly ($p \leq 0.05$). Pearson Correlation was determined between SME, BD, ER, and PD.

Results and Discussion

Proximate Composition of Binary Blends

The proximate compositions for blends were calculated from the proximate composition of the individual raw materials in the blend (Table 5.1). It can be inferred from the table that decorticated sorghum flour was had highest starch content (72.9%) and lowest crude fiber content (0.54%). Sumac flour had starch content (67.9%) and crude fiber content (0.9%). Whole sumac flour formulated with 20% bran had highest crude fiber content (1.79%). Decorticated white sorghum flour had (0.54%) crude fiber content and (1.5%) with 20% sumac bran. The proximate analysis shown that bran had highest fat content (6%), followed by sumac flour (2.7%), least for decorticated white sorghum flour (1.8%). The blends formulated with 20% bran had highest overall fat content (3.34%), followed by (2.69%) in sumac flour, (2.64%) for decorticated white sorghum flour with 20% bran, and (1.81%) without bran. The ash content is mostly dependent of fiber content. The sumac flour blends formulated with 20% bran had highest overall ash content (1.51%), (1.33%) for decorticated white sorghum flour with 20% bran, (1.0%) in sumac flour, and (0.78%) for decorticated white sorghum flour.

Specific Mechanical Energy

Lab scale extrusion

The energy input during extrusion process of cereals is dependent on resistance to flow or flow temperature (T_f) of the melt inside extruder barrel (Alavi et al., 2011). Process SME input values are represented in term of net motor load or torque in Table (5.3 and 5.4). White sorghum flour formulated with 20% sumac bran has shown highest motor load values (25%), S (100%) was 24%, SF (80%)+Bran (20%) was 22, and SF (100%) was 21 at same process conditions. The higher motor load was due to high starch content of flours. The inclusion of bran increased energy input. The starch present in blend is the primary contributor to viscosity of the blend and higher viscosity leads to higher T_f and vice versa. The increase in feed moisture resulted in lower torque input; 12% torque for 28% moisture content and 9% torque for 36% moisture (Table 5.4). Water acts as plasticize inside extruder which reduces viscosity and T_f (Chen et al. 2010). Increase in feed moisture also decreased die head pressure (data not shown). The presence of water decreases viscosity of dough in extruder, reduces conversion ratio of extruder mechanical energy into heat energy, and consequently reduces motor torque input (Akdogan, 1996; Lin et al., 2000 and Wang, 2005). Similar results were reported by Hayashi et al. (1992) and Chen et al. (2010). MG also contributed towards high lipid content in the feed blend as it acts as lubricant and decrease T_f .

Torque trends with sorghum bran/fiber addition

It can be seen from Table 5.3, that decorticated white sorghum blend with 20% sumac bran had highest torque of 35% and for sumac sorghum flour with 20% sumac bran was 32%. The proximate composition of these flours show that sumac bran had highest crude fiber content (5.3%), followed by sumac flour (0.9%) and white sorghum flour (0.5%). Fibers pose resistance to flow and higher fiber content has higher resistance to flow and thereby highest torque is observed in blend with highest fiber content.

In decorticated blends, the role of total starch present in the blends becomes important as fiber is removed and from Table 5.3, decorticated white sorghum had higher total starch content (72.9%), followed by sumac flour (starch content 67.9%), decorticated

white sorghum with 20% bran (starch content 65.11%), and sumac flour with 20% bran (starch content- 61.15%). The starch is the primary contributor of viscosity in the melt (Alavi et al., 2011). The increases in resistance to flow with increase in starch content leads to an increase in torque proportional to starch content. The torque values for decorticated white sorghum with total starch content (72.9%) was 24, followed by sumac flour with total starch content 67.95% was 21, decorticated white sorghum with 20% bran with total starch content- 65.11% was 25, and sumac flour with 20% bran with starch content- 61.15% was 22 at same feed moisture.

Pilot scale extrusion

The SME trends for pilot scale extrusion are represented in (Table 5.5). The highest energy input was 578 kJ/kg for corn flour (90%) blend and lowest SME values were for decorticated white sorghum flour formulated with 10% bran (366 kJ/kg) and 20% bran (380 kJ/kg). The higher SME in formulations was due to high starch content as compared to low starch formulations (Table 5.1). Corn flour and decorticated white sorghum flour are rich source of starch whereas whole sumac flour and condensed sumac tannins are good source of bran (fiber) and lipids and their respective incorporation into the blends impacted SME input. Increase in crude fiber content increased fat content of the blends that lowered energy inputs. Sumac bran was main source of fat (5.96%) in formulations. Effect of use of sumac bran (fiber) on SME input is represented in Figure 5.9. The 10% inclusion of sumac bran has significantly reduced energy input by 36% and 34% on 20% addition. Lipid works as a lubricant and reduces the friction between particles in the mix and between screw surfaces (Guy, 2001). The lubricating effect of oil contributes towards higher resistance to flow but decreases T_f and thereby lowers SME.

Fiber content and SME found to be inversely correlated (Figure 5.9). The SME input tends to increase with increase in fiber content of formulations. As the bran levels reached 1% SME input starts declining rapidly. The one percent fiber level could be potential threshold beyond which energy inputs adversely get effected by bran and its constituents. The possible reason could be low starch content of these formulations and

partially with increase in lipids from bran. The use of fiber effects energy input into two forms. First, high fiber bran lowered starch content of formulation which cause lowering of viscosity led to lowering energy input. Second, fibers need more energy to cook and process through extruder over starch so increase in fiber content increases SME input. In conclusion fiber and starch had more control over SME than lipids. Findings of this study clearly indicates that after a certain level extrusion energy inputs get effected by feed material compositions which affects the product attributes.

Extrudate Macrostructure

Lab scale extrusion

Expansion in extrusion process at die exit is a function of SME input and extensibility of starch matrix (Alavi et al., 2011). The extrudate expansion takes place due to the water vapor pressure inside nucleating bubbles as primary contributor for expansion. The water vapor pressure is also a function of melt temperature (Alavi et al., 2011). The higher SME input led to higher melt temperatures at die-exit resulted into greater driving force for expansion (Zhu et al. ,2010).

The values of expansion ratio (ER), and piece density (PD) are represented in Table 5.3 and 5.4. The ER was found to be higher in decorticated sorghum flour when compared to their respective whole sumac flour treatments (Table 5.3). Highest expansion was for decorticated white sorghum flour (6.71), followed by sumac flour (5.13), and decorticated white sorghum flour with 20% sumac bran (5.05), sumac flour with 20% sumac bran (3.91) (Table 5.4). The ER values were significantly different ($p < 0.05$). The higher expansion observed in decorticated sorghum flour was due to its higher torque inputs and higher starch content than whole sumac flour. Higher starch content facilitates extensible matrix results into higher expansion. The inclusion of sumac bran has clearly reduced the extrudates expansion. The 20% addition of bran into the formulation decreased the expansion ratio by 24.8% in decorticated sorghum flour formulation and 23.7% in sumac flour formulation (Figure 5.8). The presence of 1%

lipids in the form of mono-glycerides also reduced the expansion of extrudates to lowest (2.01) (Table 5.3). The lubricating effect of lipids lowered the torque, increases the melt slippage inside the extruder barrel and inhibits the sufficient pressure buildup inside the extruder leads to lower expansion (Singh et al., 2007). The fiber content acts as diluents of starch which decreases the expansion of extrudates (Bustos et al., 2011). In another study, Guy and Horne (1988) found that addition of fibers lead to fragmentation of cell membranes, and prevented the gas bubbles from expanding to their maximum capacity. The increase in moisture concentration lowered expansion ratio (Table 5.4). Water act as plasticizer inside extruder, increase in water levels decreases the viscosity and torque which led poor expansion (Figure 5.7).

Formulation with mono-glycerides had the highest piece density 0.93, followed by 0.69 for 36% feed moisture, 0.54 for 28% moisture content, 0.44 for sumac with 20% bran, 0.42 for decorticated white sorghum flour with 20% bran, 0.34 for decorticated white sorghum flour and least for sumac flour. Piece density of extrudates were significantly different ($p < 0.05$). The lower expansion reduced the volume of extrudates resulted into higher density. Piece density was a direct reflection of expansion ratio. The addition of sumac bran into sorghum flours made extrudates denser which increased the piece density for both decorticated sorghum flour and sumac flour (Table 5.3). The addition of sumac bran increased the expansion ratio of extrudates significantly ($p < 0.05$).

Pilot scale extrusion

The expansion ratio of the extrudates was affected by the type of grain and fraction used. Expansion values of extrudates produced on pilot scale extruder were represented in Table 5.5. The expansion values were ranged from 6.0 to 7.9; highest was for corn flour and lowest was for decorticated white sorghum formulated with 10% sumac bran. Whole sumac flour (99%) extrudate has shown the highest expansion among all sorghum formulations. The product expansion in extruder is directly depended of energy input and starch content (Horn, 1977). Corn meal is highly refined, composed primarily of starch (e.g. lower ash, fiber, and oil), giving maximum expansion and gelatinization. The decortication of white sorghum extruded with 10% and 20%

sumac bran decreased the expansion significantly ($p < 0.05$). This could be due to the low energy inputs because of high fat content in the bran formulations. The second reason could be the larger particle size of sumac bran. Acosta (2003) reported larger particle size of decorticated sorghum meal caused decrease in expansion upon extrusion.

Piece density was lowest for corn meal extrudates and highest for decorticated white sorghum with 20% bran. The piece density of extrudates was significantly affected by sorghum varieties and fractions in the formulation (Table 5.5). The extrudates with high expansion ration had lower piece density. Nyombaire (2007) and Fletcher et al. (1985) have all reported an inverse relation between ER and extrudate density.

Resistant Starch

Lab scale experiment

In extrusion cooking, high-temperature and high-shear forces cause a high degree of starch, degradation and gelatinization. Extrusion cooking increases starch digestibility by making it more susceptible to enzymatic hydrolysis (Holm et al., 1985), therefore, low levels of RS were expected. Breakfast cereals have low (1%-2.5%) to intermediate (2.5% to 5%) RS content depending upon process conditions (Goni et al., 1996). In agreement to literature, RS of lab scales extrudates was low as expected, and digestible starch part was high for all samples. RS fraction of lab scale extrudates are shown in Table 5.3 and 5.4, on dry matter basis. Decorticated white sorghum flour extrudate had lowest (0.45g/100g) amount of RS among all samples because it was highly-refined product. For sorghums, whole sumac and tannin extrudates had higher amounts of RS compared to white sorghum extrudates due to increased concentration of tannins (Figure 5.3). The condensed tannin products had significantly ($p < 0.05$) higher amounts of RS compared to the rest of samples. Addition of 20% condensed tannins concentration have increased RS by 40.4% in decorticated white sorghum and by

11.1% in whole sumac sorghum extrudates (Table 5.3). A positive linear correlation of $R^2=0.93$ was found with tannin concentration of the formulations (Figure 5.3).

Sorghum tannins have potential to bind amylose present in starch, and increase the RS by lowering enzymatic hydrolysis. Higher molecular weight tannins interacted with amylose and high amylose starch resulted in higher RS formation (Barros et al., 2013). The addition of sumac bran (tannins) also lowered the total starch (TS) content of the formulations (Table 5.2). Other studies have also reported increase in RS content by the inclusion of condensed sorghum tannins. Khan et al. (2013) reported increase in RS content of wheat pasta of formulating with red sorghum and white sorghum. The RS of wheat increased from 0.36% to 0.80%, 1.10% and 1.44% at 20%, 30% and 40% addition of red sorghum and to 0.64%, 0.97% and 1.16% with white sorghum addition. Similar results were found in studies by Mkandawire et al. (2013), Barros et al. (2012), Englyst et al. (2005) and Englyst et al. (2007).

Whole sumac flour formulated with 20% bran and processed at higher feed moisture conditions did not significantly ($p>0.05$) increased RS content. RS fraction was increased by 3.81% when feed moisture was increased from 22.5% to 31.4%. Similarly, RS content was increased by 0.15% at 38.4% feed moisture (Table 5.4). The reason could be decrease low shear/ energy inputs at high feed moisture (Table 5.4); shear rate is represented in term of motor load. High shear in extrusion process reduces tannins concentrations, especially higher molecular weight procyanidins (tannins) (Awika et al., 2003). Higher molecular weight procyanidins have shown higher tendency to form RS content. Therefore increase in feed moisture content lowered shear rate which resulted in higher RS (Figure 5.1). The possible reason for insignificant increase in RS content could be loss of higher molecular weight procyanidins. Tannin percentages based on theoretical calculations of each raw ingredient are presented in Table 5.2. Khan et al. (2013) have reported lower RS fraction formation in extruded wheat pasta over bread formulated with sorghum tannins. Addition of 1% mono-glycerides lowered motor load but it didn't raise RS content (Table 5.3). The possible

reason could be unavailability of amylose for tannins to interact because of amylose-lipid complex formation.

A comparison of RS before and after extrusion is represented in Figure 5.5. Resistant starch of formulations before extrusion were calculated theoretically based on RS fractions of individual flours. Sorghum flour RS content was 0.72g/100g before, sumac flour was 17.74g/100g before extrusion, RS was 0.45g/100gm for sorghum flour and 0.83 g/100gm sumac flour in expanded extrudates. Similarly, RS values of all formulations were reduced to less than one percent post extrusion (Figure 5.5). Awika et al. (2003) reported 76% loss of RS content (from 20.51g/100 to 4.86g/100gm) in extrusion processing. The processing conditions effect overall distribution of tannins (procyanidins) polymers units and their content in food. Extrusion processing of sorghum tannins breakdown higher molecular weight polymers to their lower molecular weight constituents.

Pilot Scale extrusion

RS values of pilot scale extrudates ranged from 0.07g/100gm to 0.74g/100gm on dry matter basis (Table 5.5). Corn flour had lowest amount of RS in products. Corn flour extrudate had highest amount of total starch among all samples because it was a highly-refined product. Whole sumac flour extrudates had highest RS may be because of highest tannins concentration (2.18%). Tannin concentrations of each raw ingredient were calculated theoretically using Awika et al. (2003) published sorghum tannin results (Table 5.2). Tannin have shown a high propensity to interact with amylose starch in RS formation and lowered starch enzymatic digestion. Barros et al. (2013); reported 0.152% of RS when normal starch was cooked with tannins and 0.069% RS for precooked starch cooked with tannins in auto clave cooking method. Also, reported RS content of 0.391%-high tannin and 0.331%-sumac tannins with high amylose. The RS content increased from 0.437% to 0.472% when pure tannins were oven cooked with high amylose. Similarly, 0.425%-high tannin and 0.367%-sumac tannin with high amylose starch in oven cooking method. RS of cooked sorghum with tannins was in the range of 3.6% and 15.4% (Mkandawire et al., 2013). Englyst et al. (2005, 2007)

reported 2% to 10% RS in cooked whole grain sorghum. Similarly, RS content of 3% to 14% was reported Austin et al. (2012) in sorghum bread formulated with sorghum bran. In this study, we found a very strong positive linear correlation ($R^2=0.64$) of RS formation with tannin concentrations of formulations (Figure 5.4). RS contents of samples were significantly ($p<0.05$) different. Similarly, addition of 10% sumac bran (tannins) in decorticated white sorghum increased RS content to 0.42g/100gm by 15% and 20% bran addition increase to 0.53g/100g by 26% (Figure 5.2). Results of this study agree with Barros et al. (2012) and Khan et al. (2013). The 49% addition of corn flour into whole sumac sorghum has lowered RS by 10% due to drop in (1.1%) tannin concentrations (Table 5.4).

Drop in RS content post extrusion was also found in pilot scale extruded samples. The range of RS fraction in raw material was between 0.45g/100g to 17.56g/100g. A significant drop in RS content was observed post extrusion; values ranged from 0.07g/100g to 0.74g/100g (Figure 5.6). High SME input in pilot scale extrusion could have destroyed polymerized tannins molecules which were no longer able to prevent enzymatic hydrolysis of starch. Similar, results of low RS contents post processing were reported by Awika et al. (2003), Khan et al. (2013) and Goni et al. (1996).

Conclusion

In conclusion first study demonstrates specific finding on resistant starch formation with condensed tannins in extrusion process. The study also determines effect of (motor load) energy input on resistant starch formation in expanded snack. Sorghum condensed tannins are more effective in interacting with total starch content through hydrophobic and hydrogen bonding, significantly increased RS content. The data demonstrates that sorghum and sorghum fractions rich in procyanidins can be processed into various cereals based foods and retains a significant amount of levels of RS content that may have functional properties. This study also confirms addition of whole sumac sorghum and condensed tannins into expanded snack at all incorporation levels effectively enhanced RS content; of possible benefits in diets to help prevention

of chronic eased related to oxidative stress such as type-2 diabetes mellitus and for improved intestinal health. Additional studies are now required to evaluate consumer acceptability.

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Table 5.1: Proximate analysis of raw material used in this study.

Samples	Crude protein (%)	Crude fiber (%)	Fat (%)	Ash (%)	Total starch (%)
Corn flour	5.0±0.0	<0.2±0.0	1.9±0.0	0.5±0.0	78.2±0.2
Sorghum flour	9.7±0.0	0.5±0.1	1.8±0.1	0.8±0.0	72.9±0.0
Sumac flour	0.2±0.0	0.9±0.1	2.7±0.3	1.0±0.1	68.0±0.4
Sumac bran	0.8±0.0	5.3±0.0	6.0±0.1	3.6±0.1	34.0±0.4

Corn flour- degermed yellow corn flour, Sorghum flour- decorticated white sorghum flour, Sumac flour- whole sumac flour.

Table 5.2: Tannins, resistant starch, and total starch in raw ingredients (on dry mass basis).

Samples	Tannins (g/100g)	RS (g/100g)	Total Starch (g/100g)
Corn Flour	0.00	0.47±0.02	65.2±0.2
Sorghum flour	0.00	0.72±0.09	62.1±0.0
Sumac flour	0.022	17.74±0.19	60.8±0.4
Sumac bran	0.058	15.93±0.68	24.8±0.4

RS-resistant starch, tannins values are calculated theoretically based on the findings of Awika et al., 2004. Tannins for whole sumac flour ≈ 0.022 g/100gm, for condensed tannin bran ≈ 0.058 g/100.

Table 5.3: Process moisture, net motor load, expansion ratio, piece density, resistant starch of lab scale extrudates produced with different sorghum varieties and sumac bran.

Treatments	Moisture (%)	Net motor load (%)	ER	PD (g/cm ³)	RS (g/100g)	
					Aft. Ext	Bef. Ext
SF(100%)	22.2±0.1 ^c	24±1.4 ^a	6.7±0.8 ^a	0.34±0.5 ^e	0.45±0.01 ^c	0.72
SF(80%)+Bran(20%)	21.6±0.3 ^c	25±0.0 ^a	5.1±0.7 ^b	0.42±0.0 ^d	0.63±0.08 ^{bc}	3.76
SuF(100%)	21.5±0.0 ^c	21±0.0 ^a	5.1±1.2 ^b	0.27±0.1 ^e	0.81±0.02 ^{ba}	17.74
SuF(80%)+Bran(20%)	21.7±0.4 ^c	22±0.7 ^a	3.9±0.9 ^c	0.44±0.1 ^d	0.90±0.02 ^a	17.37
SuF(79%)+Bran(20%)+MG(1%)	21.8±0.3 ^c	11±1.4 ^b	2.0±0.4 ^d	0.93±0.2 ^a	0.79±0.02 ^{ba}	17.20

Moisture is represented in percentage, ER-expansion ratio, PD- Piece density, SF-decorticated white sorghum flour, SuF- whole sumac flour, Bran- sumac bran, RS-resistant starch as dry matter basis (material), Aft. Ext.- after extrusion, Bef. Ext. – before extrusion (predicted values based on theoretical calculation).

Table 5.4: Process moisture, motor load, expansion ration, piece density, resistant starch for lab scale extrudates processed at different process moisture conditions.

Treatments	Moisture	Net Motor	ER	PD	RS (g/100g)	
	(%)	load (%)		(g/cm ³)	Aft. Ext	Bef. Ext
SuF(80%)+Bran(20%)	21.7±0.4 ^c	22±0.7 ^a	3.9±0.9 ^c	0.44±0.1 ^d	0.90±0.02 ^a	17.37
SuF(80%)+Bran(20%)	31.4±0.9 ^b	12±2.8 ^b	2.7±0.2 ^d	0.54±0.0 ^c	0.94±0.10 ^a	17.37
SuF(80%)+Bran(20%)	38.4±0.1 ^a	9±2.1 ^b	2.4±0.3 ^d	0.69±0.0 ^b	0.94±0.00 ^a	17.37

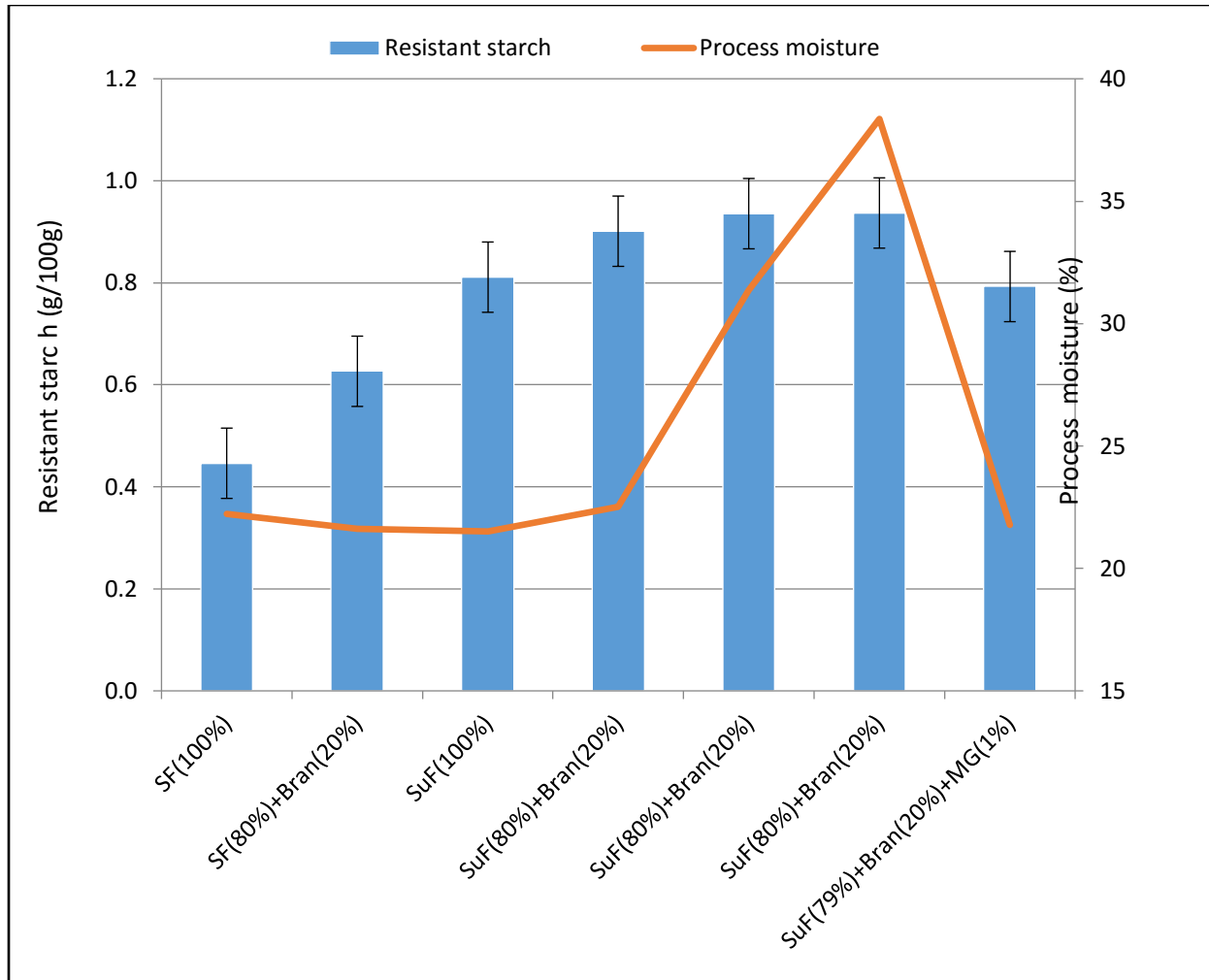
Moisture is represented in percentage, ER-expansion ratio, PD- Piece density, SF-decorticated white sorghum flour, SuF- whole sumac flour, Bran-sumac bran, RS-resistant starch as dry matter basis (material), Aft. Ext.- after extrusion, Bef. Ext. – before extrusion (predicted values based on theoretical calculation).

Table 5.5: SME, process moisture, expansion ration, piece density and resistant starch of pilot scale extrudates.

Treatments	Process	SME (KJ/KG)	ER	PD (g/cm ³)	RS (g/100g)	
	Moisture				Aft. Ext	Bef. Ext
CF(99%)+Salt(1%)	19.0±1.2 ^{ab}	578±12 ^a	7.9±0.1 ^a	0.43±0.0 ^c	0.07±0.01 ^e	0.45
SF(99%)+Salt(1%)	20.3±1.5 ^a	543±27 ^{ba}	7.3±0.3 ^{ab}	0.65±0.0 ^b	0.37±0.02 ^d	0.71
SuF(99%)+Salt(1%)	17.7±0.8 ^b	573±21 ^a	7.7±0.2 ^a	0.57±0.0 ^{bc}	0.74±0.04 ^a	17.56
CF(49%)+SuF(50%)+Salt(1%)	20.8±1.0 ^a	500±17 ^b	7.0±0.4 ^{ab}	0.46±0.0 ^c	0.67±0.02 ^a	9.09
CF(49%)+SF(50%)+Salt(1%)	21.5±1.2 ^a	506±33 ^{ba}	7.5±0.3 ^a	0.60±0.0 ^b	0.48±0.04 ^{cb}	0.58
SF(89%)+Bran(10%)+ Salt(1%)	19.9±0.6 ^a	366±24 ^c	6.0±0.9 ^b	0.69±0.1 ^{ab}	0.42±0.02 ^{cd}	2.23
SF(79%)+Bran(20%)+ Salt(1%)	19.5±0.8 ^a	388±36 ^c	6.4±0.7 ^b	0.79±0.1 ^a	0.53±0.01 ^b	3.75

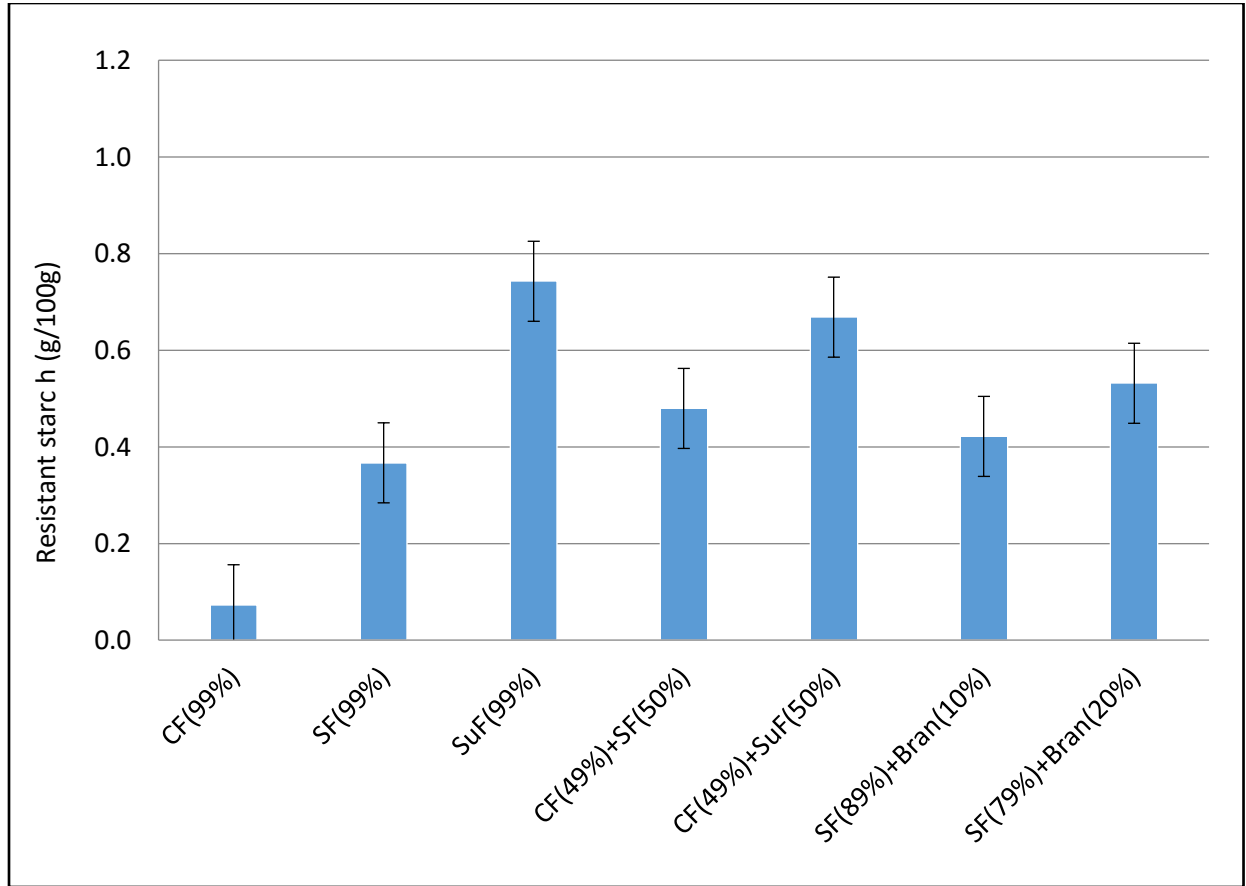
CF- corn flour, SF-decorticated white sorghum flour, SuF- whole sumac flour, Bran- sumac bran, process moisture in percentages, SME- Specific mechanical energy, ER- expansion ratio, PD- piece density, RS-resistant starch as dry matter basis (material), Aft. Ext.- after extrusion, Bef. Ext. – before extrusion (predicted values based on theoretical calculation).

Figure 5.1: Resistant starch content of lab scale extrudates (on dry matter basis).



SF-decorticated white sorghum flour, SuF- sumac flour, Bran- sumac bran, MG- mono-glycerides.

Figure 5.2: Resistant starch content of pilot scale extrudates (on dry matter basis).



SF-decorticated white sorghum flour, SuF- sumac flour, Bran- sumac bran, MG- mono-glycerides. The 1% residual is salt that makes the formulations add to 100%.

Figure 5.3: Correlation between resistant starch content and tannin concentration for lab scale extrudates.

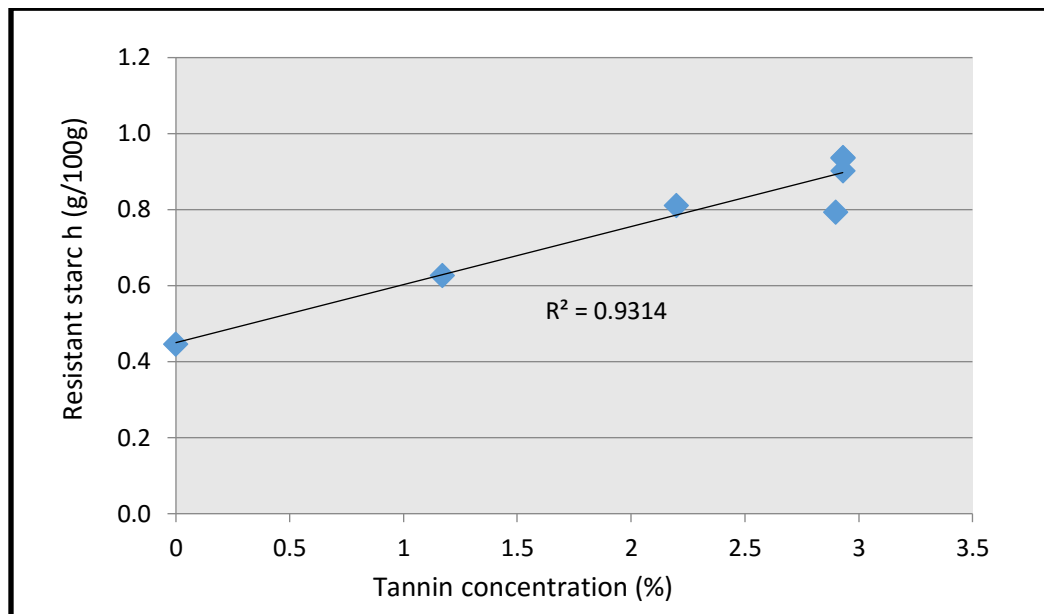


Figure 5.4: Correlation between resistant starch content and tannin concentration for pilot scale extrudates.

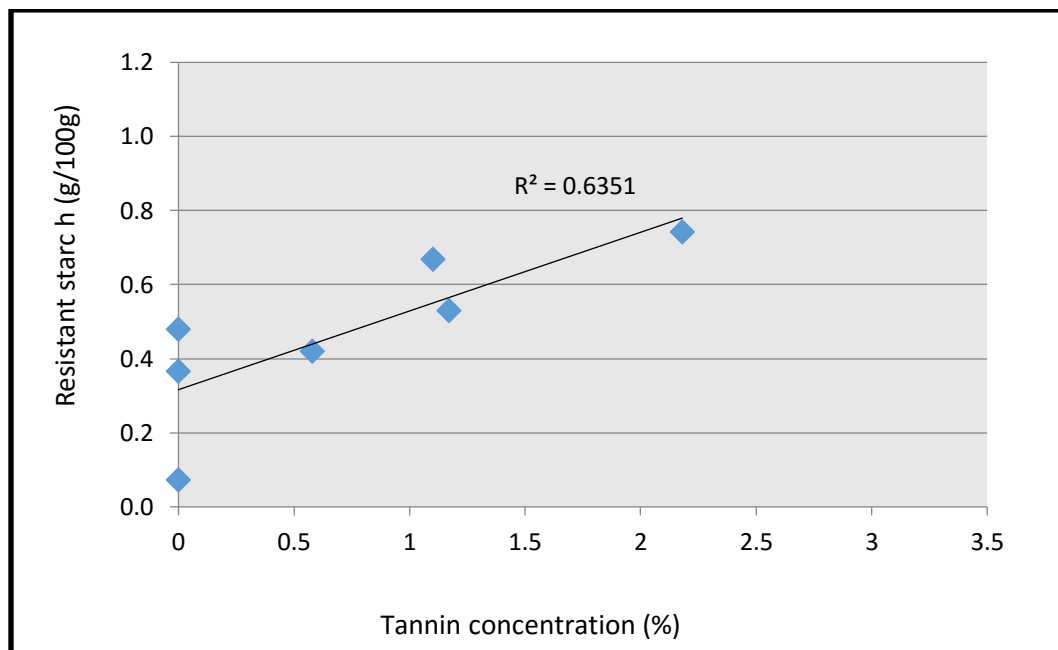
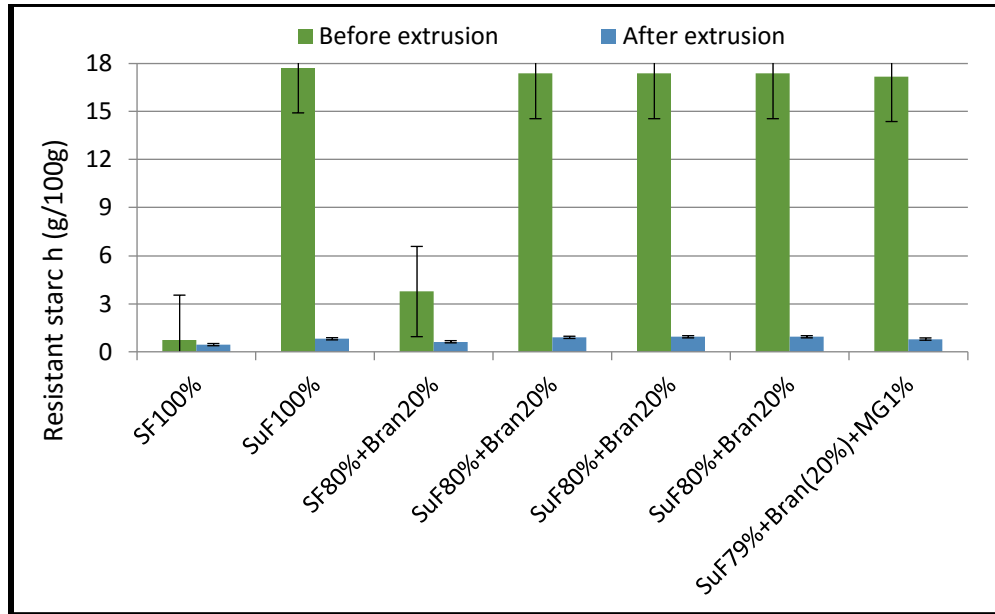
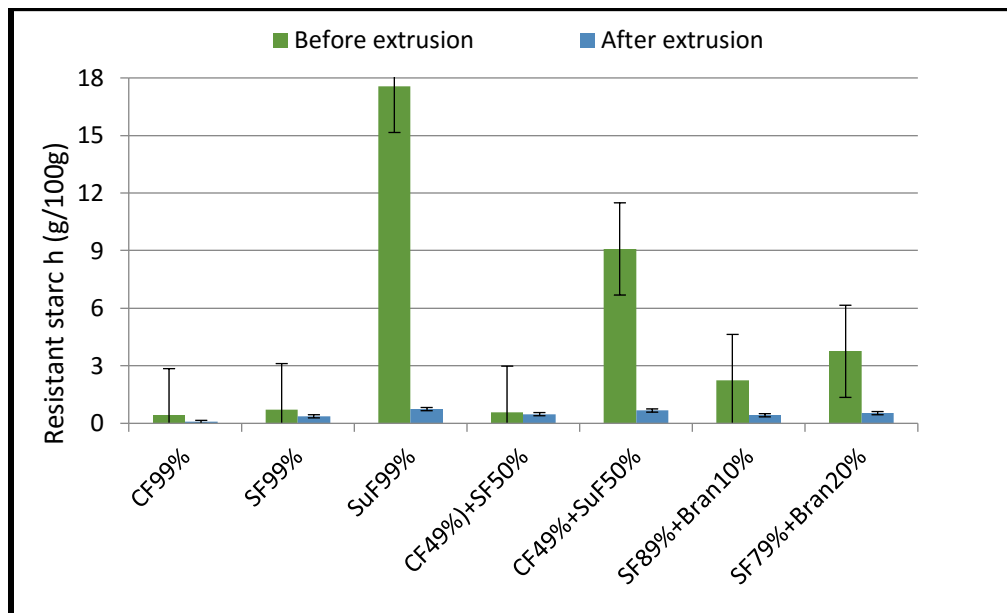


Figure 5.5: Resistant starch content of lab scale extrudates before and after extrusion processing.



SF-decorticated white sorghum flour, SuF- sumac flour, Bran- sumac bran, MG- mono-glycerides.

Figure 5.6: Resistant starch content of pilot scale extrudates before and after extrusion processing.



CF- Corn flour, SF-decorticated white sorghum flour, SuF- sumac flour, Bran- sumac bran, MG- mono-glycerides.

Figure 5.7:Effect of moisture content on average expansion ratio of lab scale extrudates formulated with sumac sorghum (80%) and sumac bran (20%) processed at different feed moistures.

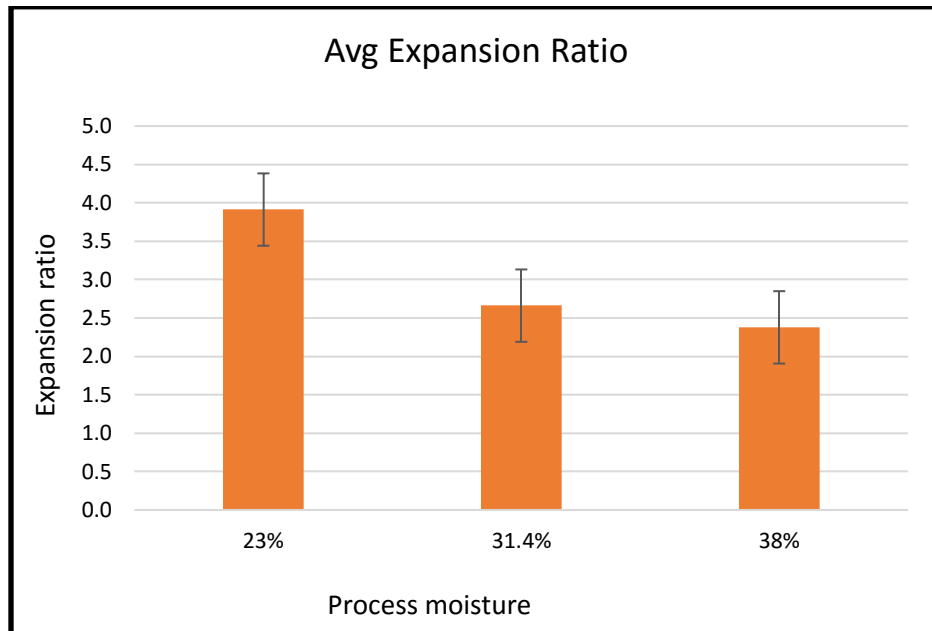
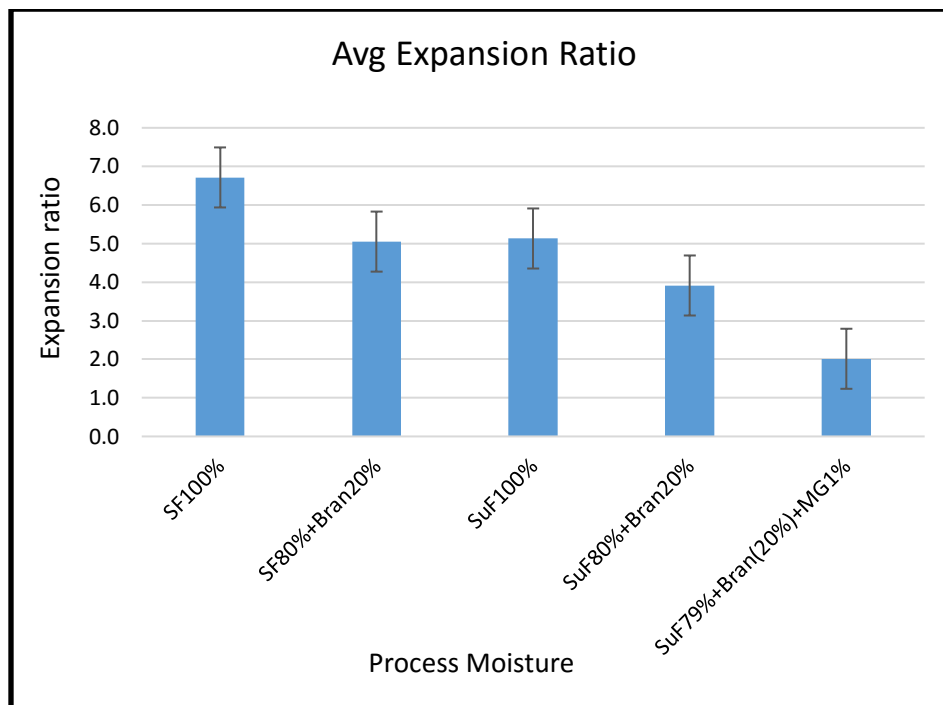
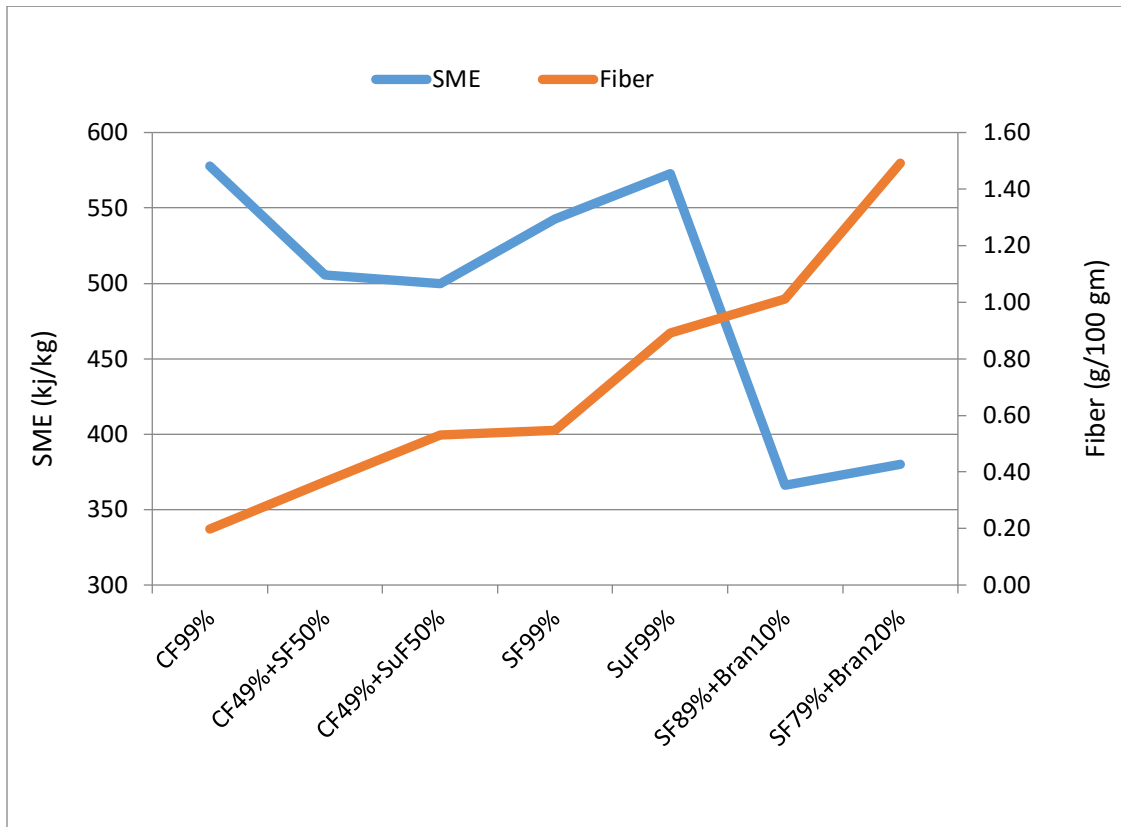


Figure 5.8:Effect of different sorghum varieties and bran on average expansion ratio of lab scale extrudates.



SF-decorticated white sorghum flour, SuF- sumac flour, Bran- sumac bran, MG- mono-glycerides.

Figure 5.9: SME variation with fiber content of the formulations in pilot scale extrusion.



CF- Corn flour, SF-decorticated white sorghum flour, SuF- sumac flour, Bran- sumac bran, MG- mono-glycerides.

Chapter 6 - Conclusion and Future Work

Teff have potential to produce high quality pasta with addition of texture enhancers. Bench top extruded teff flour pasta was of superior quality with low cooking loss, high water absorption and good textural properties. Proximate analysis has shown that higher protein content, fiber content and mineral content makes teff pasta a nutritionally excellent product. High lipid content of millet pasta degrades its physico-chemical characteristics and shelf life.

Sorghum pasta was of top quality than teff and millet on pilot scale extrusion, with lower cooking losses, higher water absorption and textural properties. The optimum concentration of mono-glycerides was found to be 1% for sorghum pasta. The optimum process moisture was found to be 48% for sorghum pasta. The addition of native corn starch did not help in lowering cooking losses for teff and millet flour significantly. Millet pasta produced on pilot scale disintegrated during cooking reflects extremely poor quality.

The residence time significantly decreased by increase in in-barrel moisture content during sorghum pasta processing. Low in-barrel moisture decreased feed material residence time inside extruder barrel also increased RTD spread. Addition of lipids in the form of mono-glycerides into formulation has significantly increased mean residence time and residence time spread. The flow was moving toward a mixed flow with increase of lipid concentration and toward plug flow with decrease of lipid concentration. The addition of sumac bran into formulations lowered the SME input during processing of expanded snack. Sumac bran inclusion raised lipid content that increased lubrication in extrusion process resulted into lower energy input. Sumac sorghum based expanded snack had higher resistant starch content over white sorghum extrudates. Whole sumac flour and sumac bran formulation increased resistant starch in extrudates but it remained lower than 1%. Addition of sumac sorghum varieties doesn't significantly increase the resistant starch of extrudates.

Future work

Results obtained in this study opens new horizons in processing research of sorghum, millet and teff. The study provides a detailed information to the world of food science where every day new cereals products are becoming part of our daily life.

Experiment to analyze the addition of gluten in teff and millet based formulations to see its effect on strengthening starch-protein matrix. The optimized in-barrel moisture and lipid level results of this can be applied to other cereals for product development. Pasta produced in this study still not subjected to sensory study. The consumer study can give great information on consumer insights such as product taste, liking, perceptibility etc. Additionally, shelf life study of sorghum, millet and teff pasta can also be included in future work.

APPENDIX A - Figures

Figure A.1: Cooking loss at 0.5% MG and with two levels of 28% amylose corn starch of teff and millet pasta.

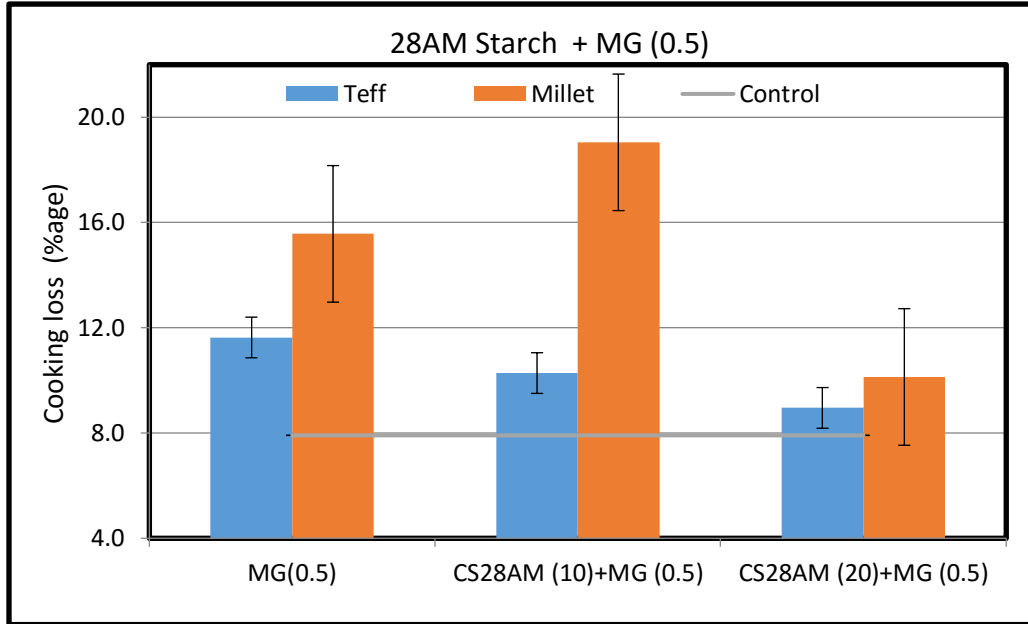


Figure A.2: Cooking loss at 1% MG and with two levels of 28% amylose corn starch of teff and millet pasta.

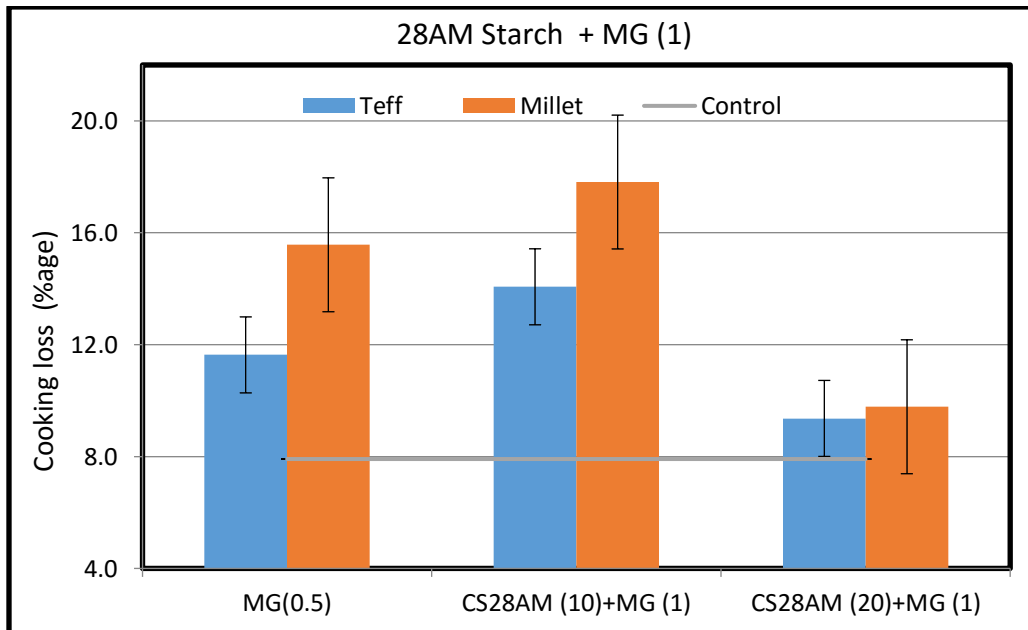


Figure A.3: Cooking loss at 0.5% MG and with two levels of 55% amylose corn starch of teff and millet pasta.

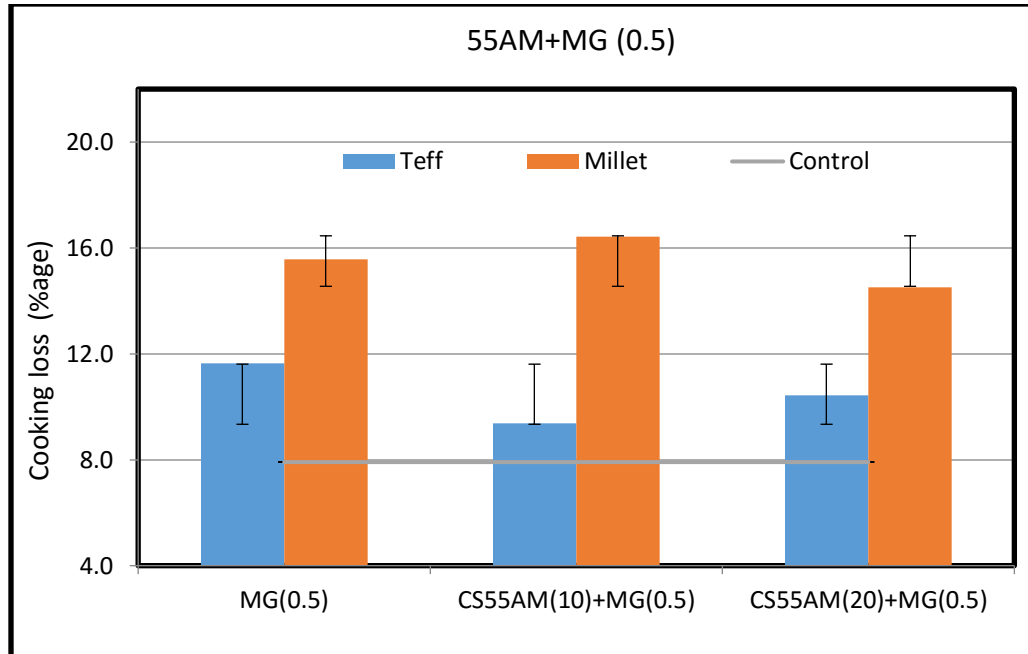


Figure A.4: Cooking loss at 1% MG and with two levels of 55% amylose corn starch of teff and millet pasta.

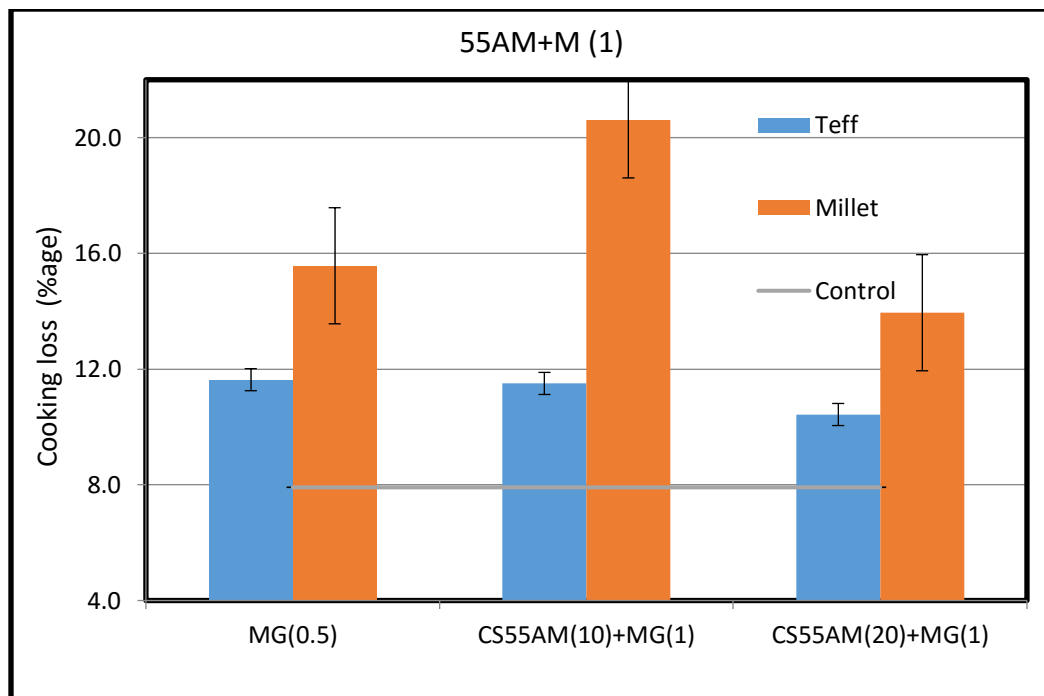


Figure A.5: Cooking loss at 10% addition of 28% amylose corn starch of teff and millet pasta.

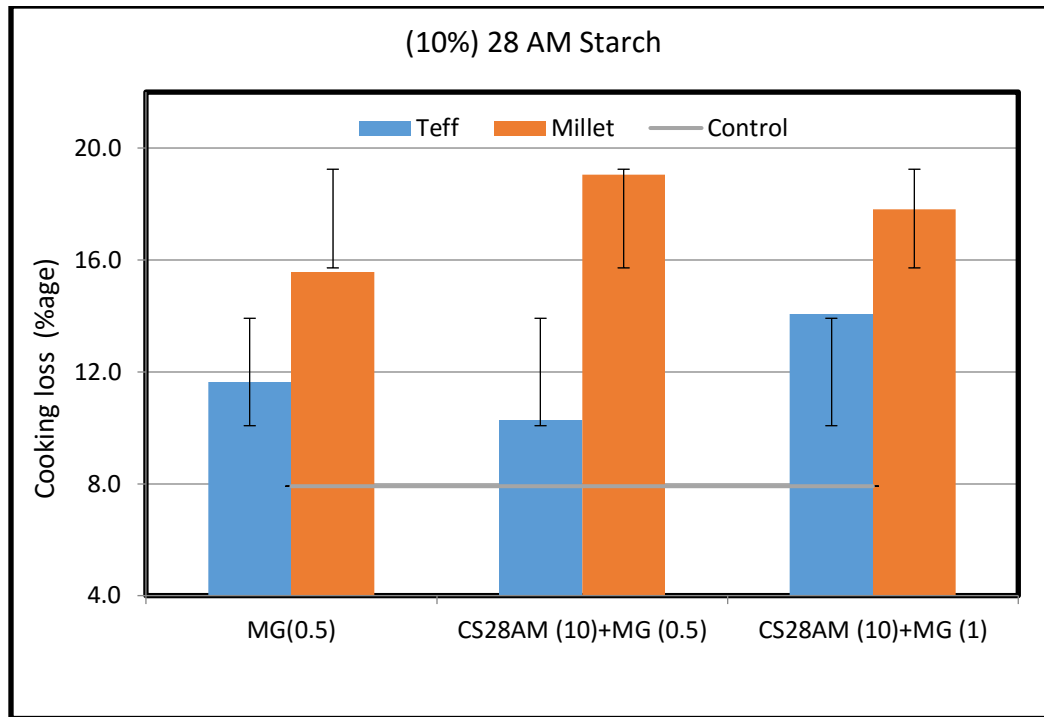


Figure A.6: Cooking loss at 20% addition of 28% amylose corn starch of teff and millet pasta.

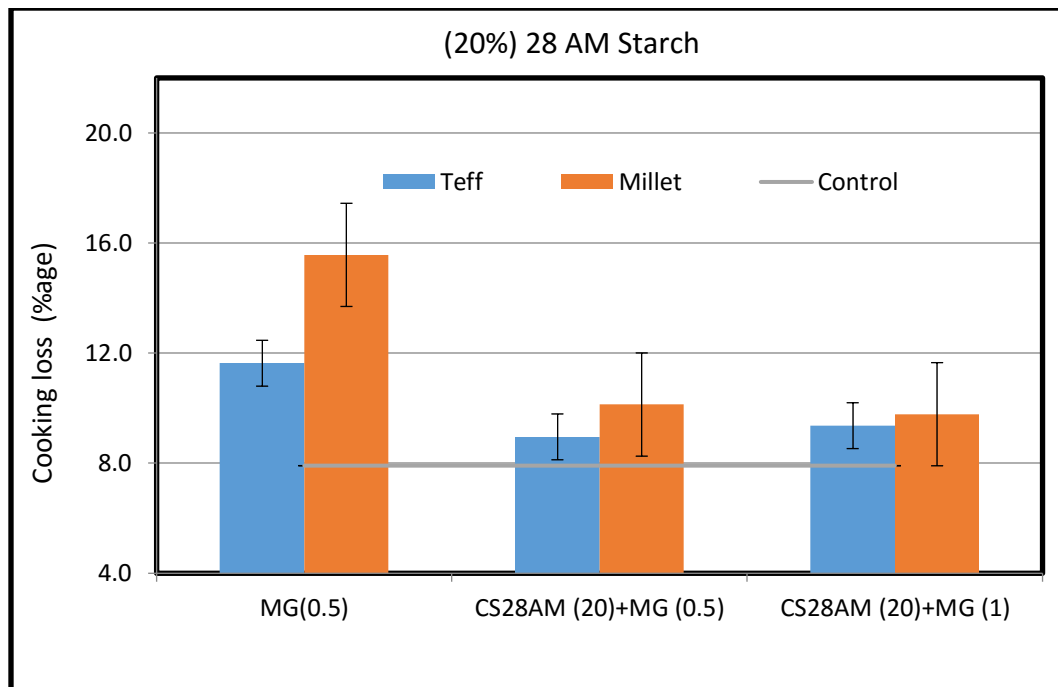


Figure A.7: Cooking loss at 10% addition of 55% amylose corn starch of teff and millet pasta.

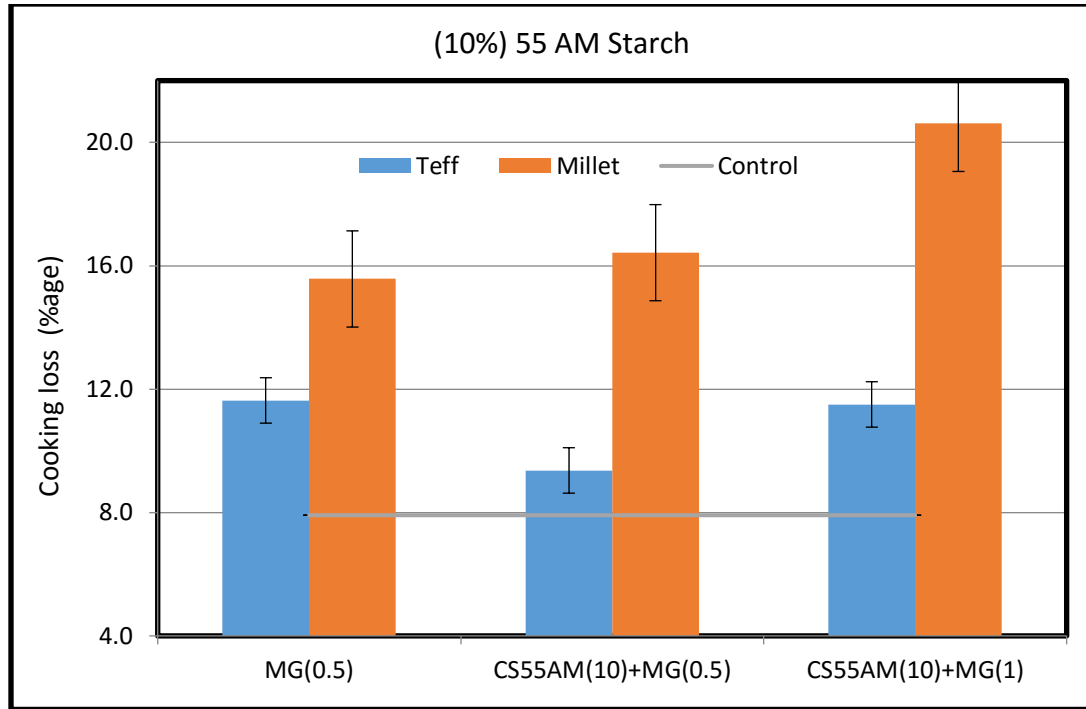


Figure A.8: Cooking loss at 20% addition of 55% amylose corn starch of teff and millet pasta.

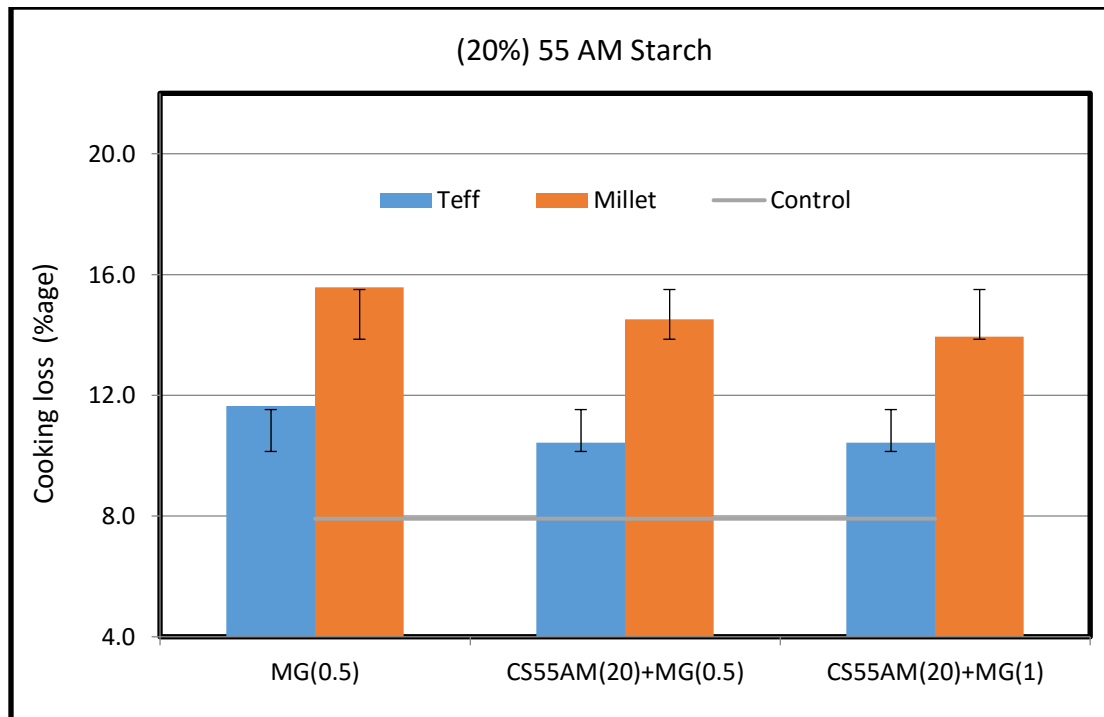


Figure A.9: Water absorption at 0.5% MG and with two levels of 28% amylose corn starch of teff and millet pasta.

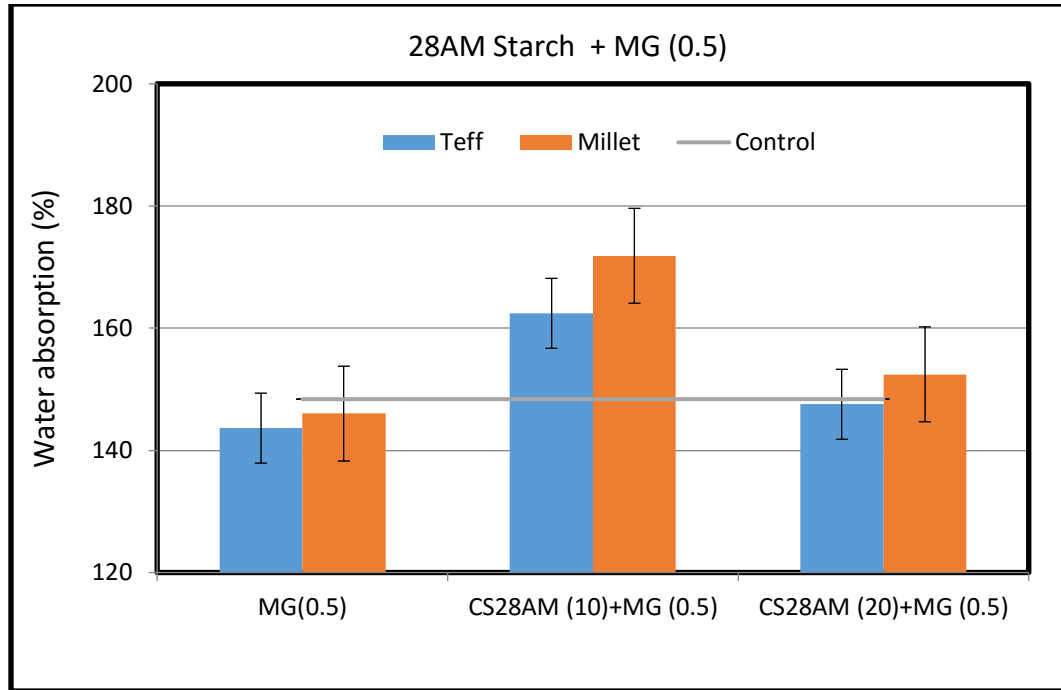


Figure A.10: Water absorption at 1% MG and with two levels of 28% amylose corn starch of teff and millet pasta.

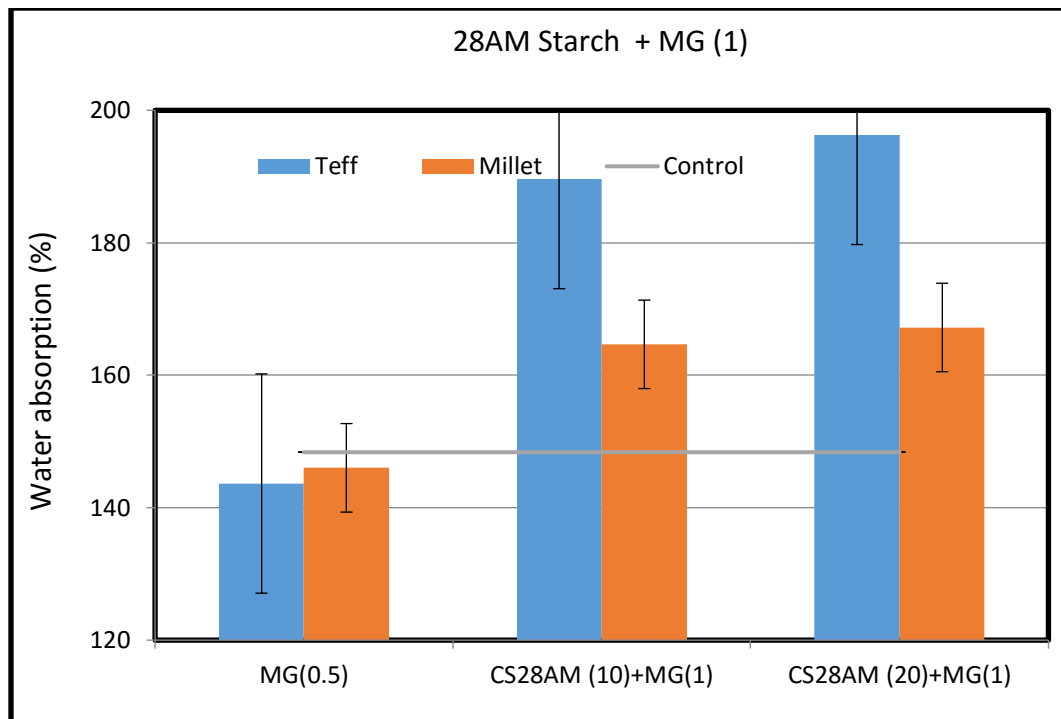


Figure A.11: Water absorption at 0.5% MG and with two levels of 55% amylose corn starch of teff and millet pasta.

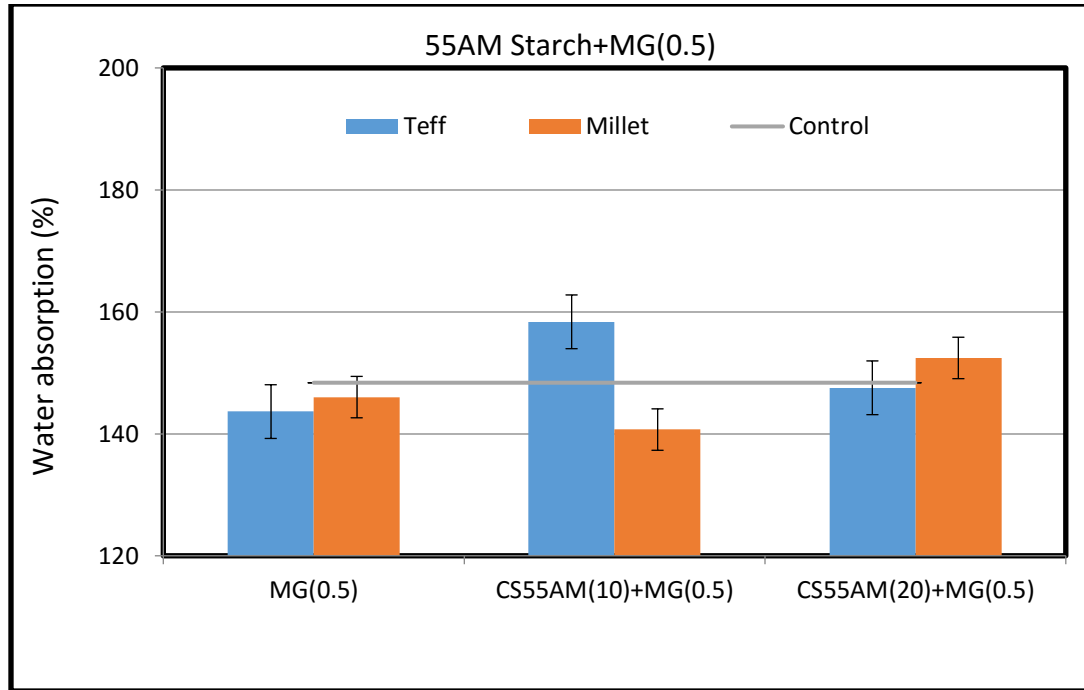


Figure A.12: Water absorption at 1% MG and with two levels of 55% amylose corn starch of teff and millet pasta.

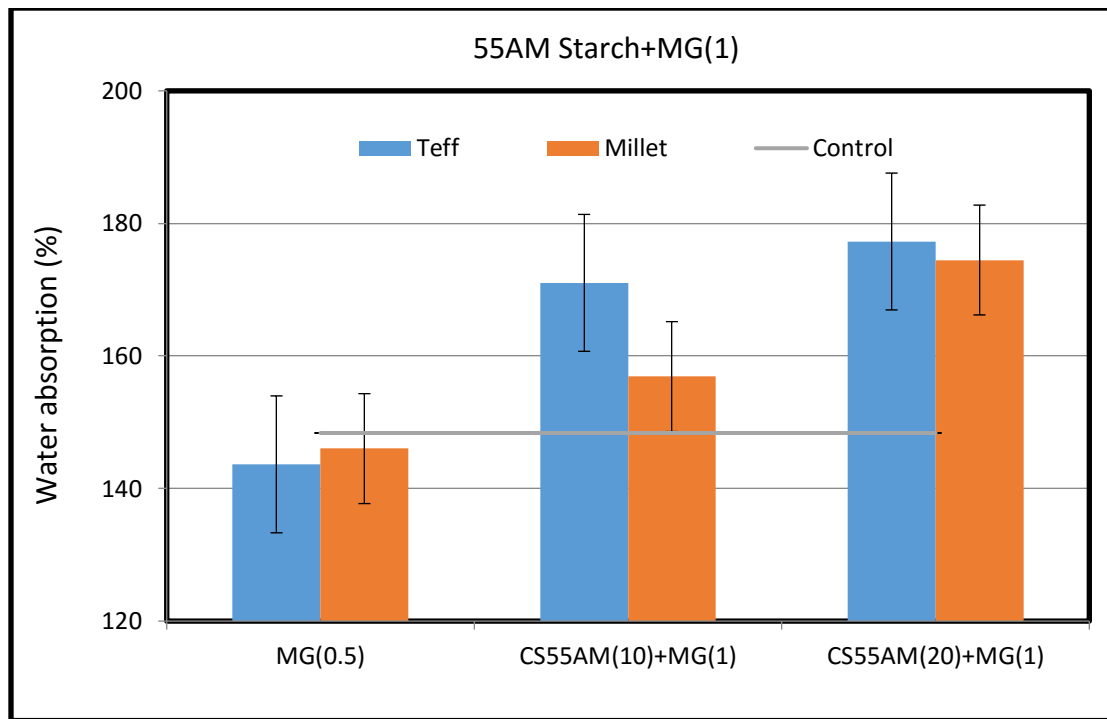


Figure A.13: Water absorption at 10% addition of 28% amylose corn starch of teff and millet pasta.

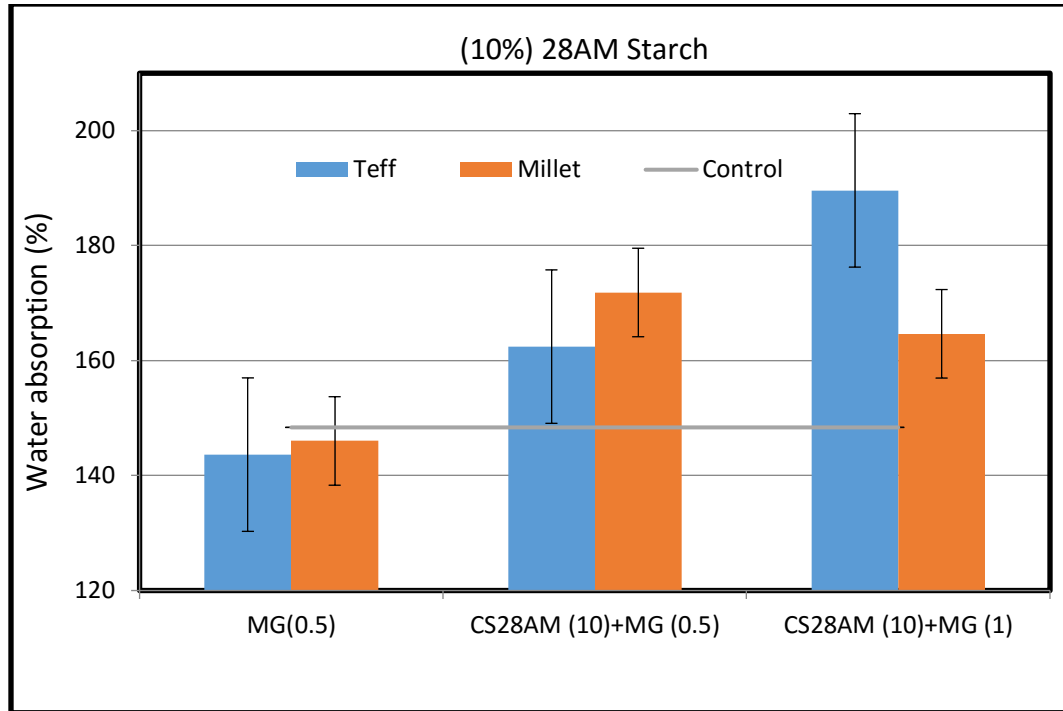


Figure A.14: Water absorption at 20% addition of 28% amylose corn starch of teff and millet pasta.

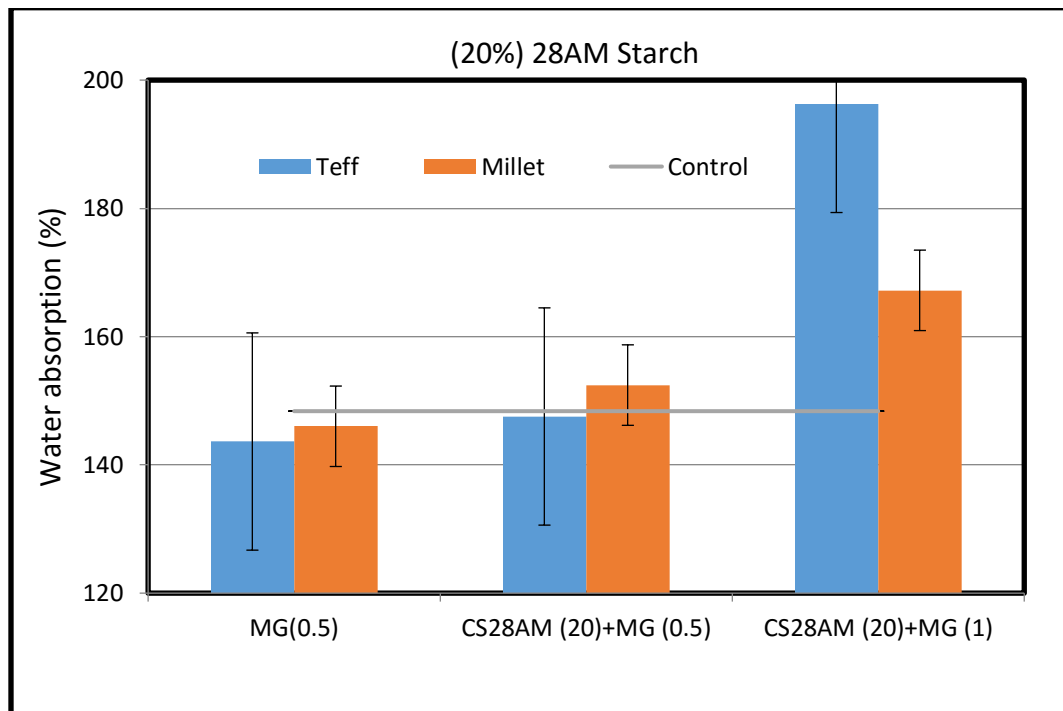


Figure A.15: Water absorption at 10% addition of 55% amylose corn starch of teff and millet pasta.

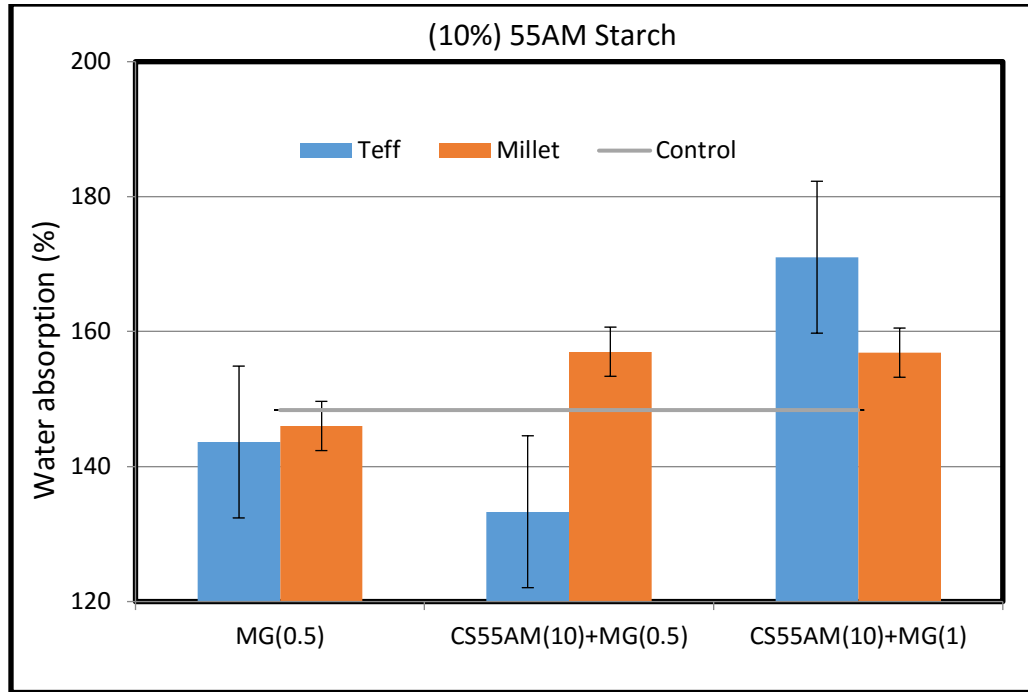
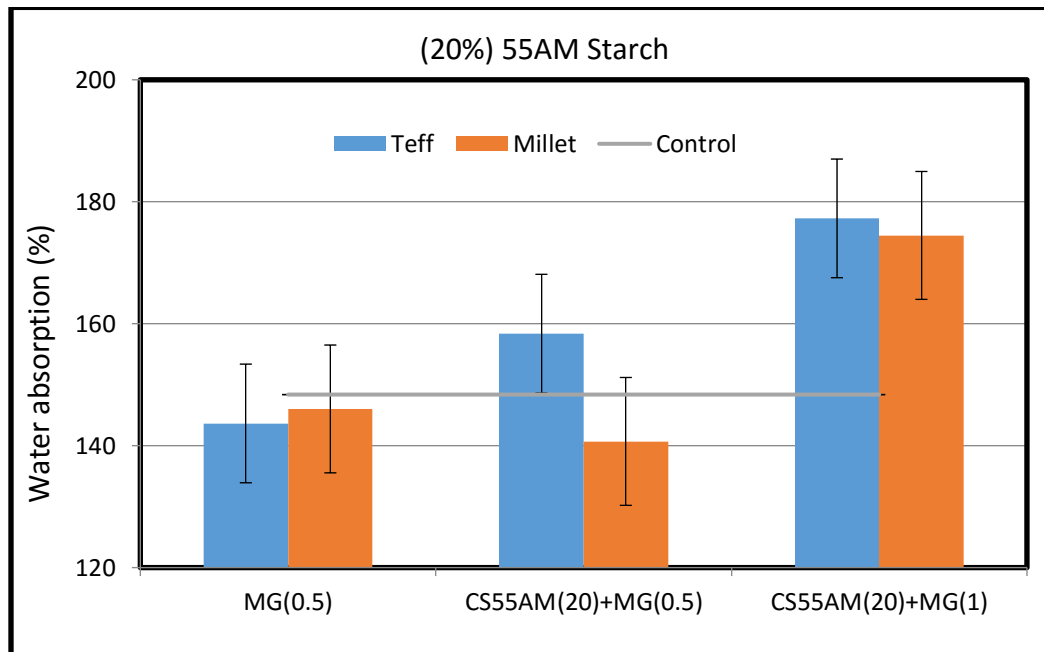


Figure A.16: Water absorption at 20% addition of 55% amylose corn starch of teff and millet pasta.



RVA pasting curves of different teff flour based raw material and blends used in experiment.

Figure A.17: Pasting curve of raw teff flour, teff and semolina with 0.5% mono-glycerides.

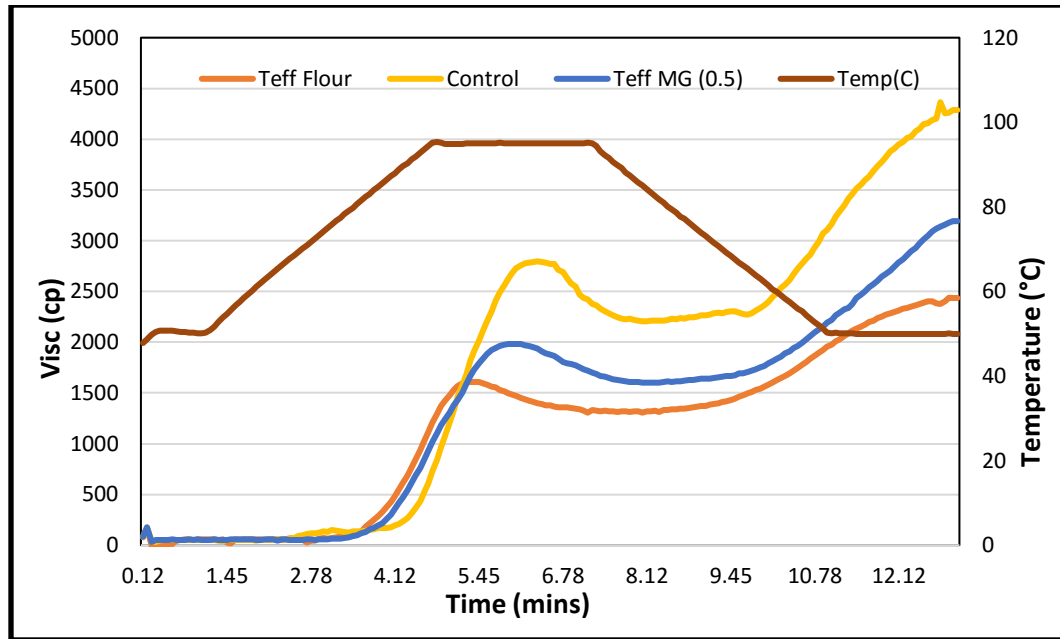


Figure A.18: Pasting curve of raw teff flour blends formulated mono glycerides and 55% amylose corn starch.

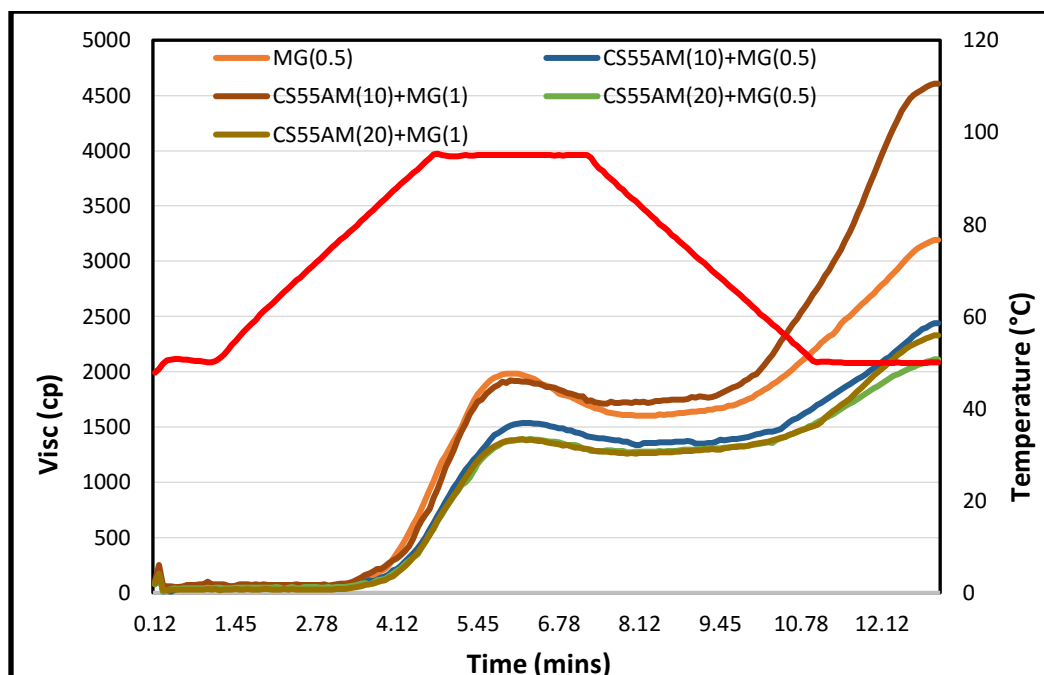


Figure A.19: Pasting curve of raw teff flour blends formulated mono glycerides and 28% amylose corn starch.

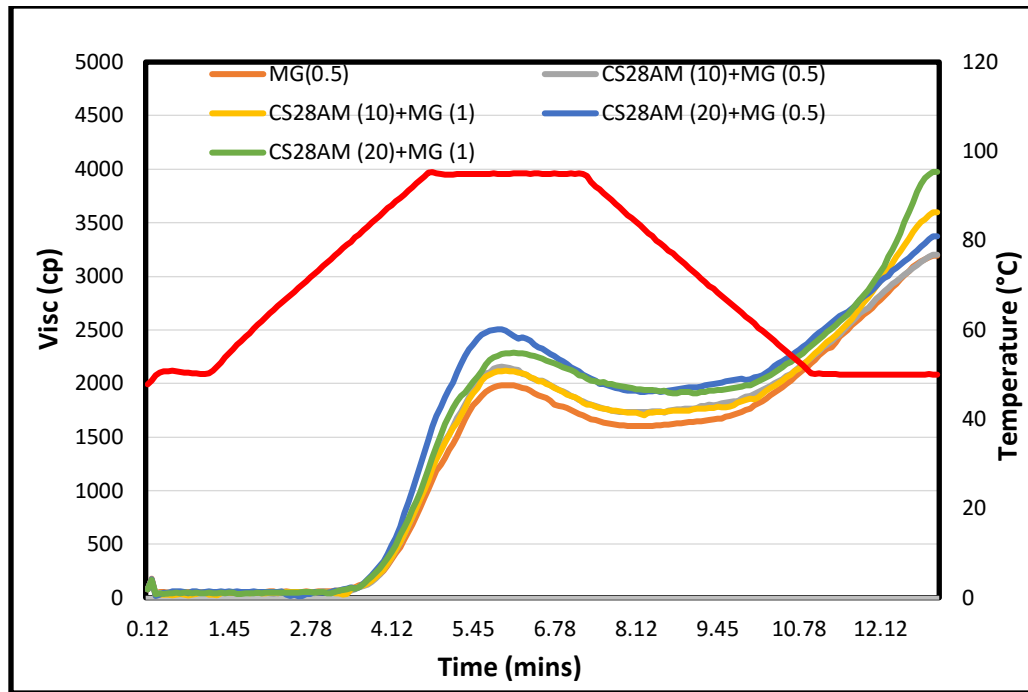


Figure A.20: Pasting curve of extruded precooked pasta products teff flour, teff flour with 0.5% mono-glycerides, semolina with 0.5% mono-glycerides.

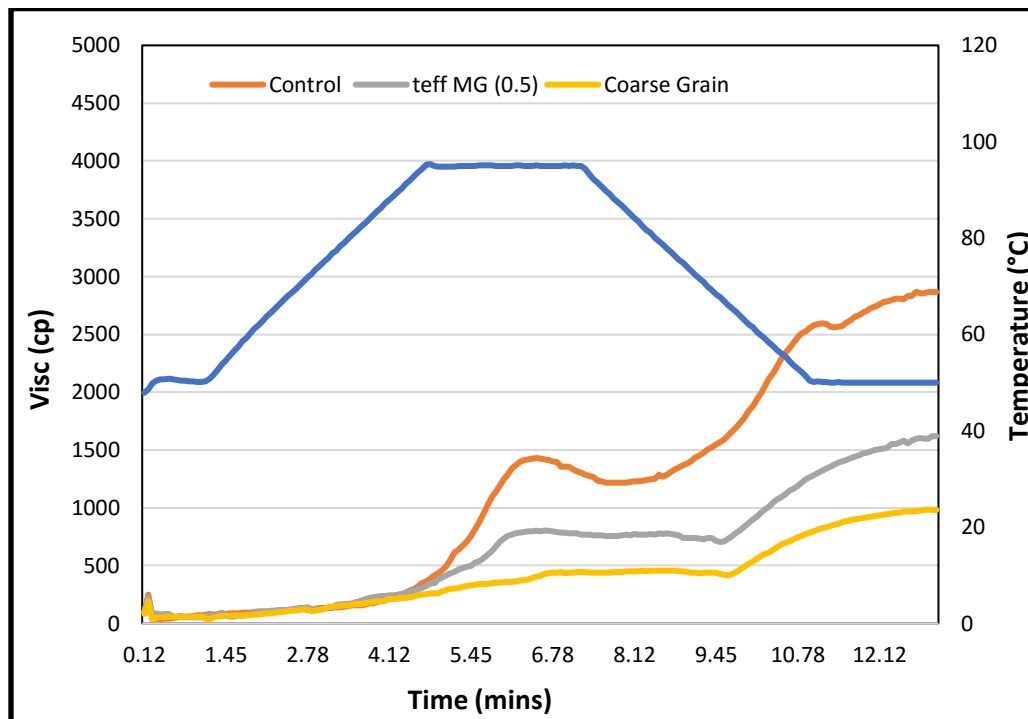


Figure A 21: Pasting curve of extruded teff pasta blends formulated mono glycerides and 55% amylose corn starch.

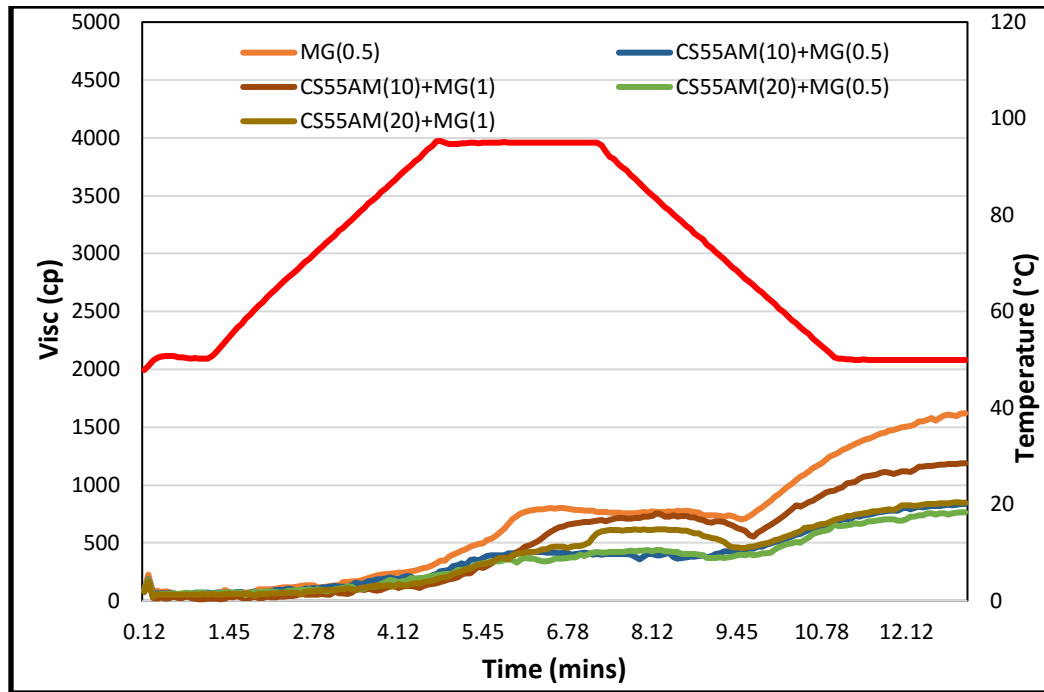
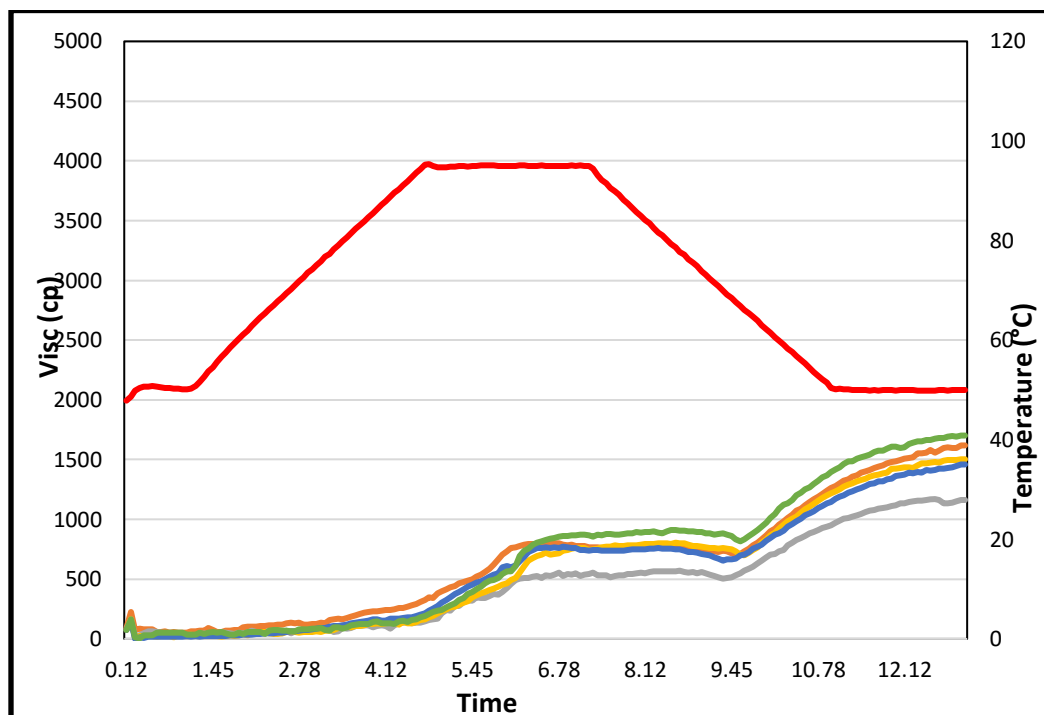


Figure A.22: Pasting curve of extruded teff pasta blends formulated mono glycerides and 28% amylose corn starch.



RVA pasting curves of different millet flour based raw material and blends used in experiment.

Figure A.23:Pasting curve of raw millet flour, millet flour with 0.5% mono-glycerides, semolina with 0.5% mono-glycerides.

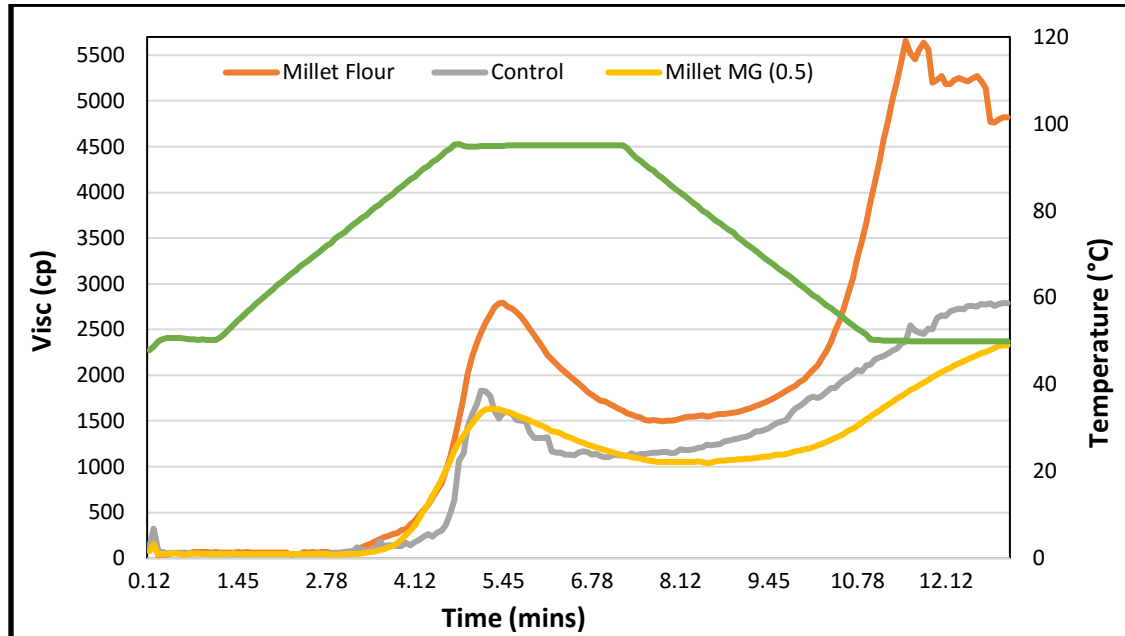


Figure A 24:Pasting curve of raw millet flour blends formulated mono glycerides and 55% amylose corn starch.

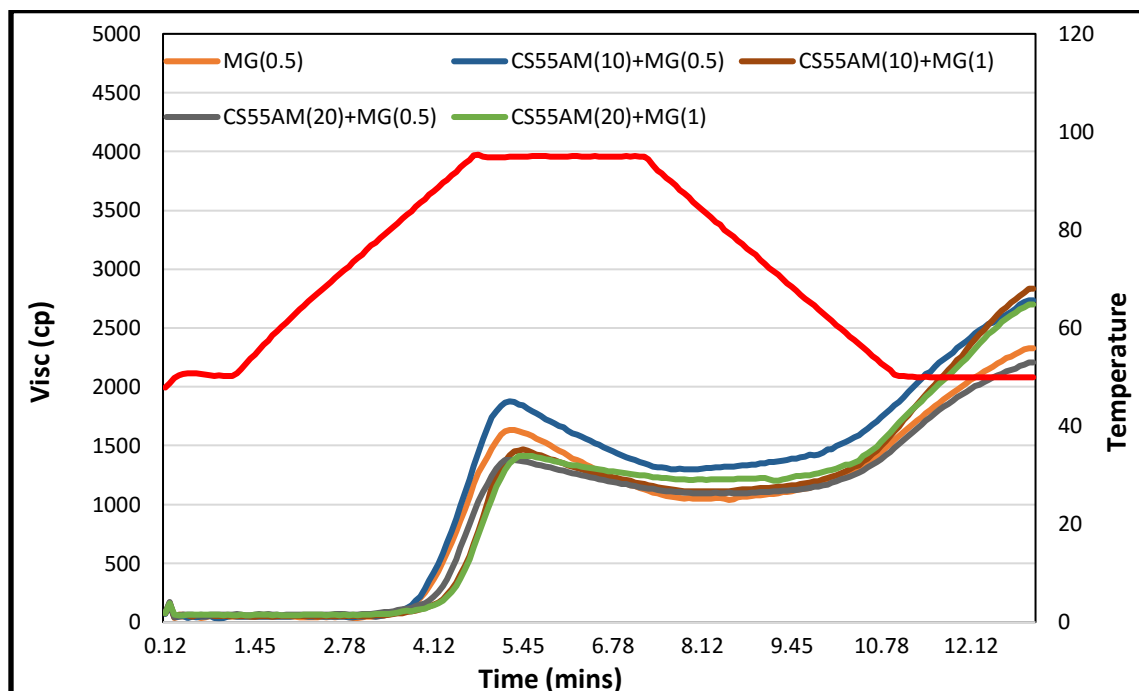


Figure A.25: Pasting curve of raw millet flour blends formulated mono glycerides and 28% amylose corn starch.

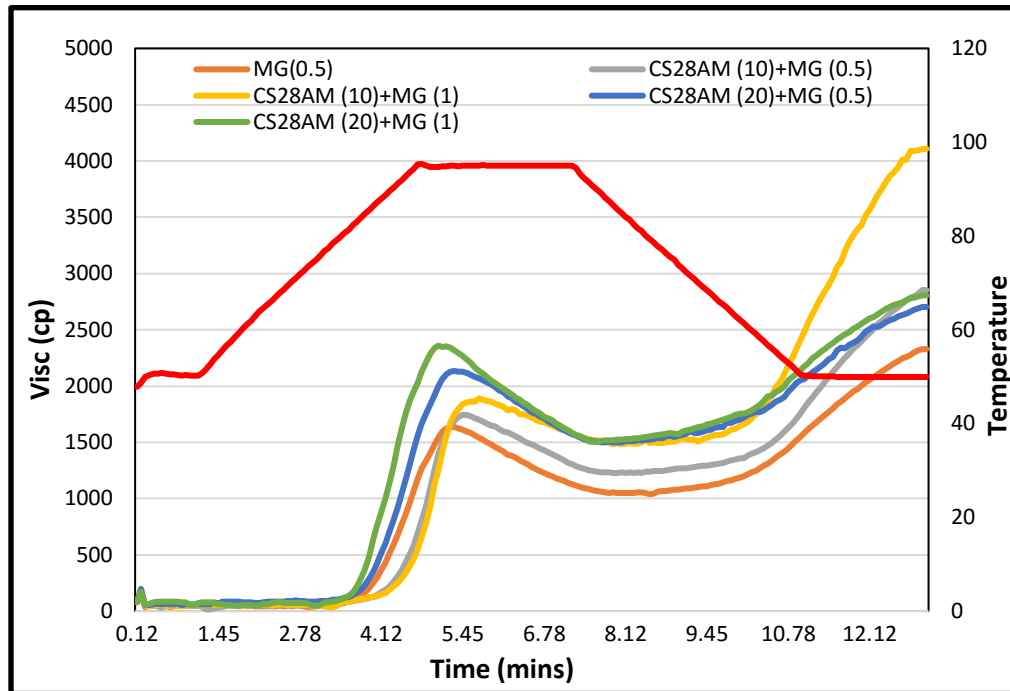


Figure A.26: Pasting curve of extruded precooked millet flour, millet flour with 0.5% mono-glycerides and semolina with 0.5% mono-glycerides.

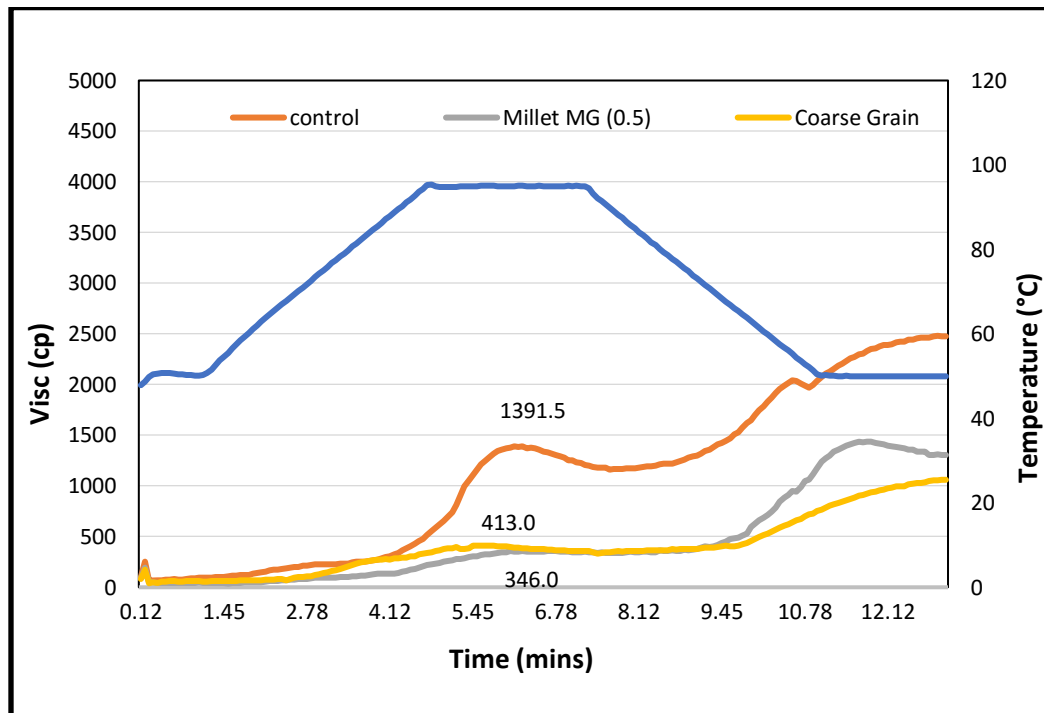


Figure A.27: Pasting curve of extruded millet pasta blends formulated mono glycerides and 55% amylose corn starch.

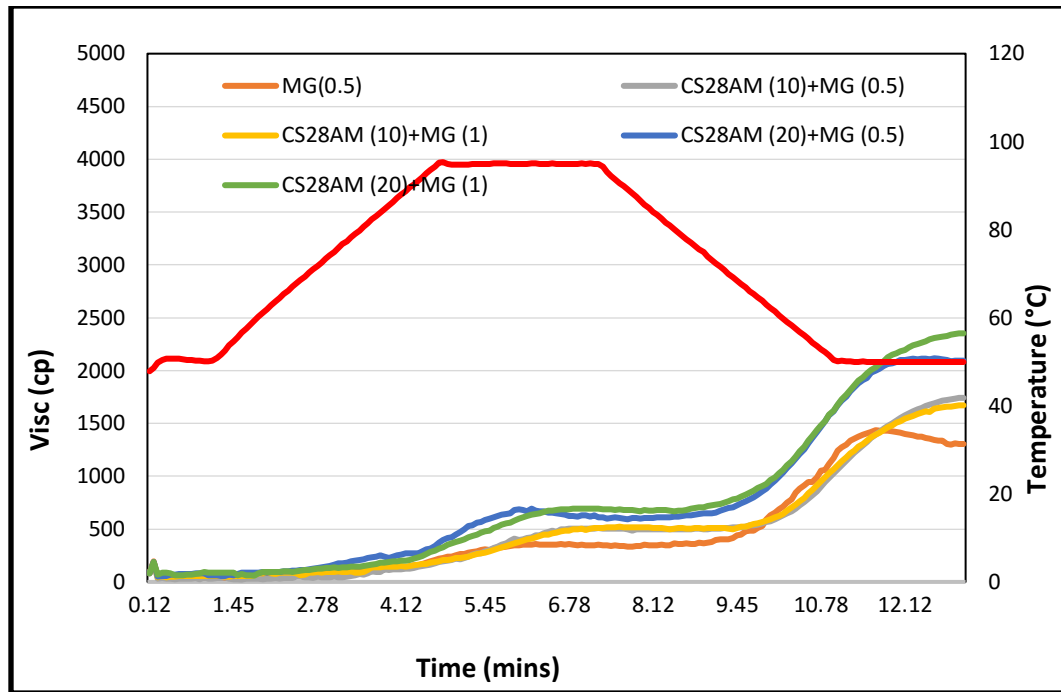
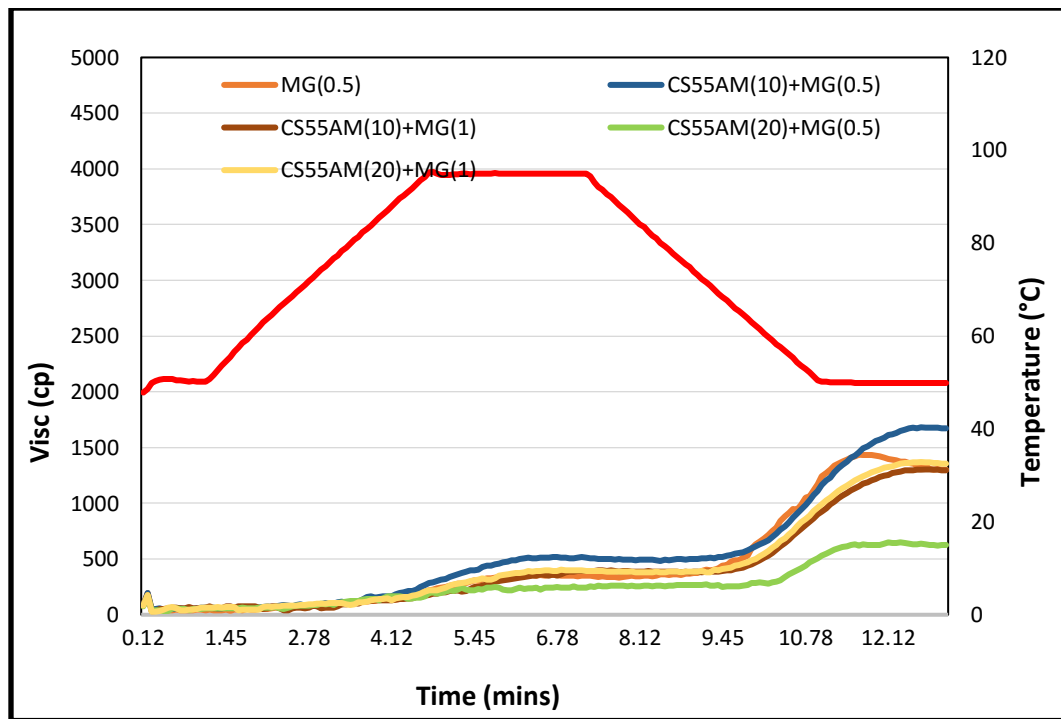


Figure 28: Pasting curve of extruded millet pasta blends formulated mono glycerides and 55% amylose corn starch.



Differential scanning calorimeter (DSC) curves of different raw flours

Figure A.29: DCS curves of raw semolina.

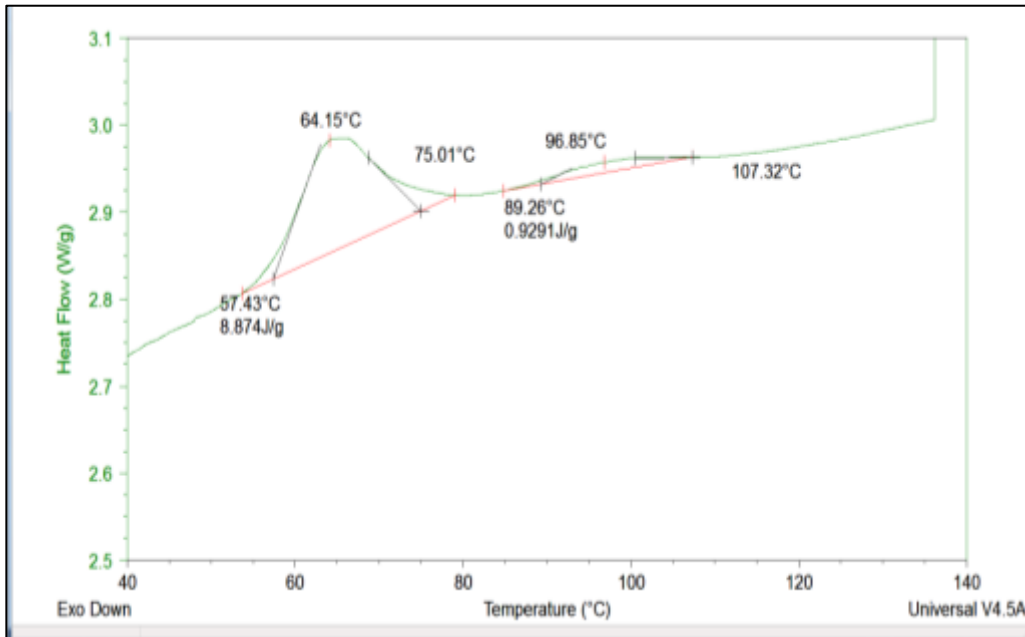


Figure A.30: DCS curves of raw sorghum flour.

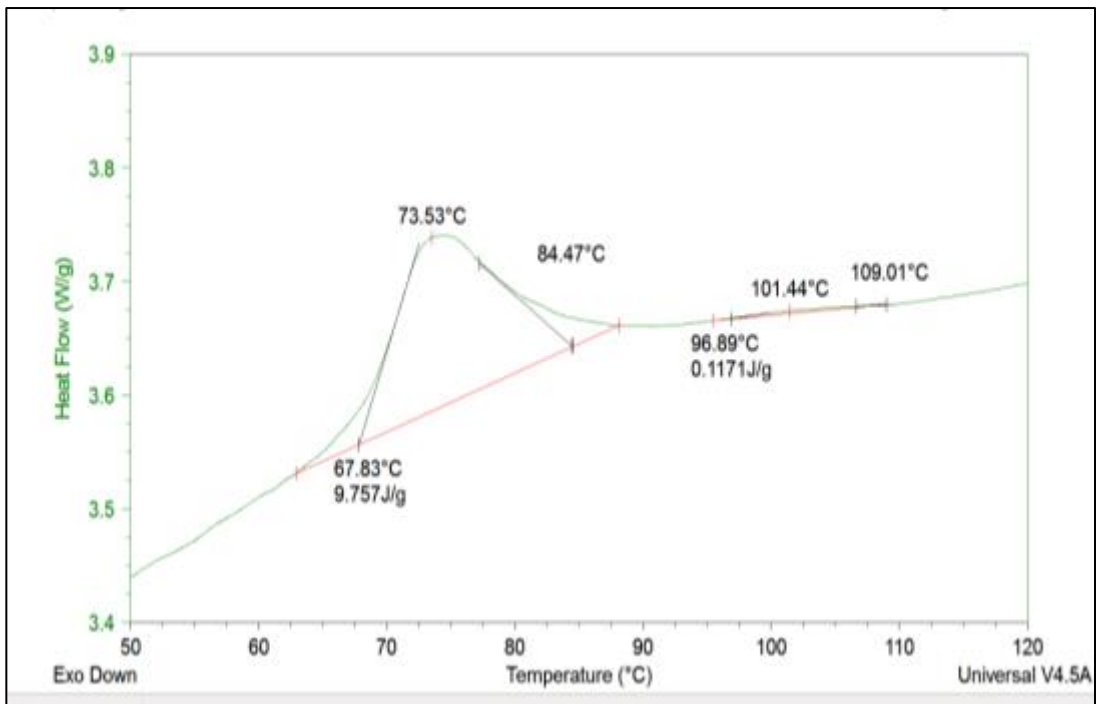


Figure A.31:DCS curves of raw teff flour.

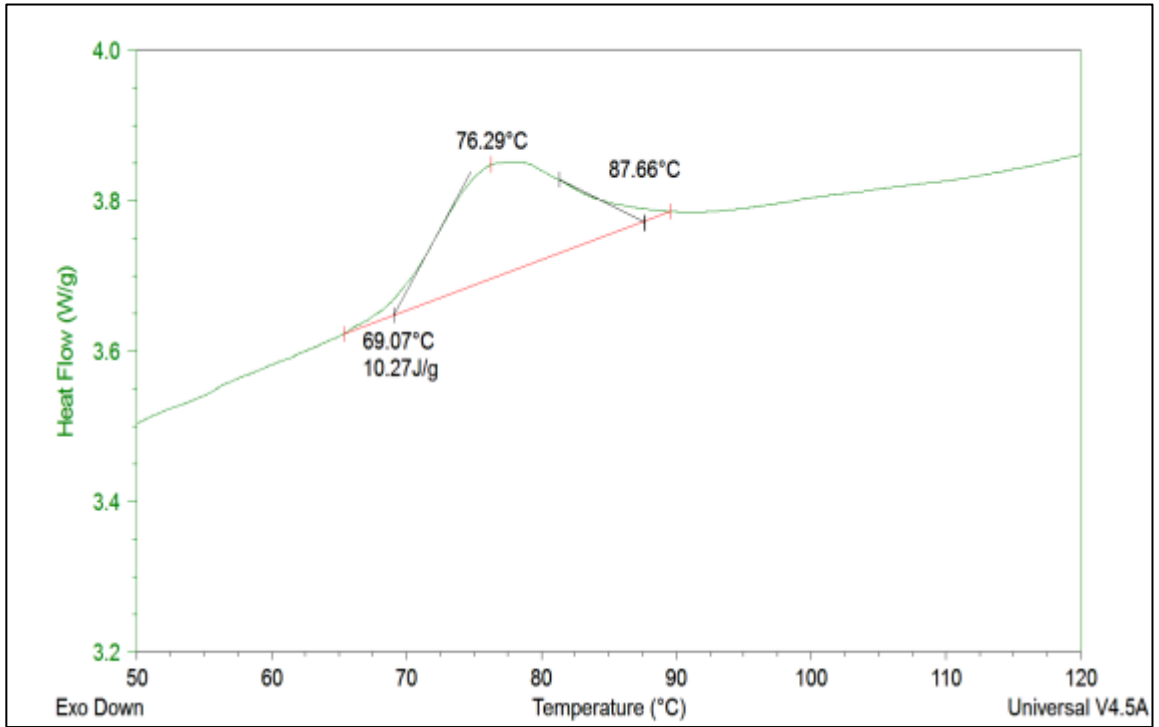


Figure A.32:DCS curves of raw millet flour.

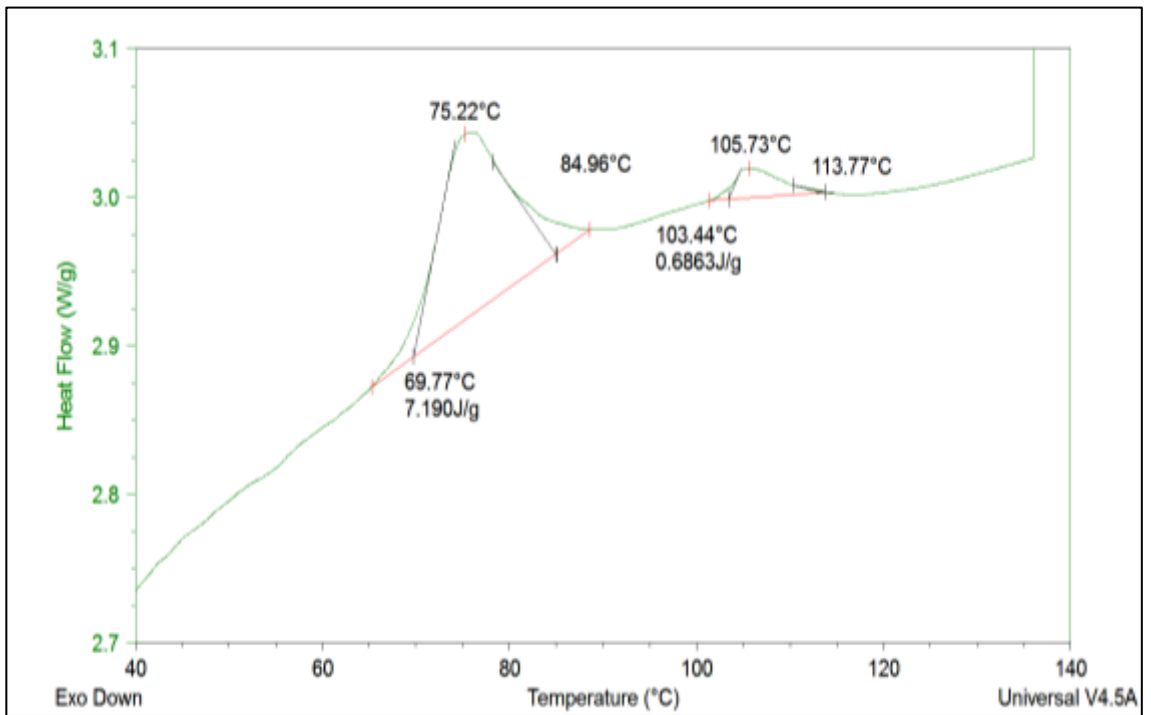
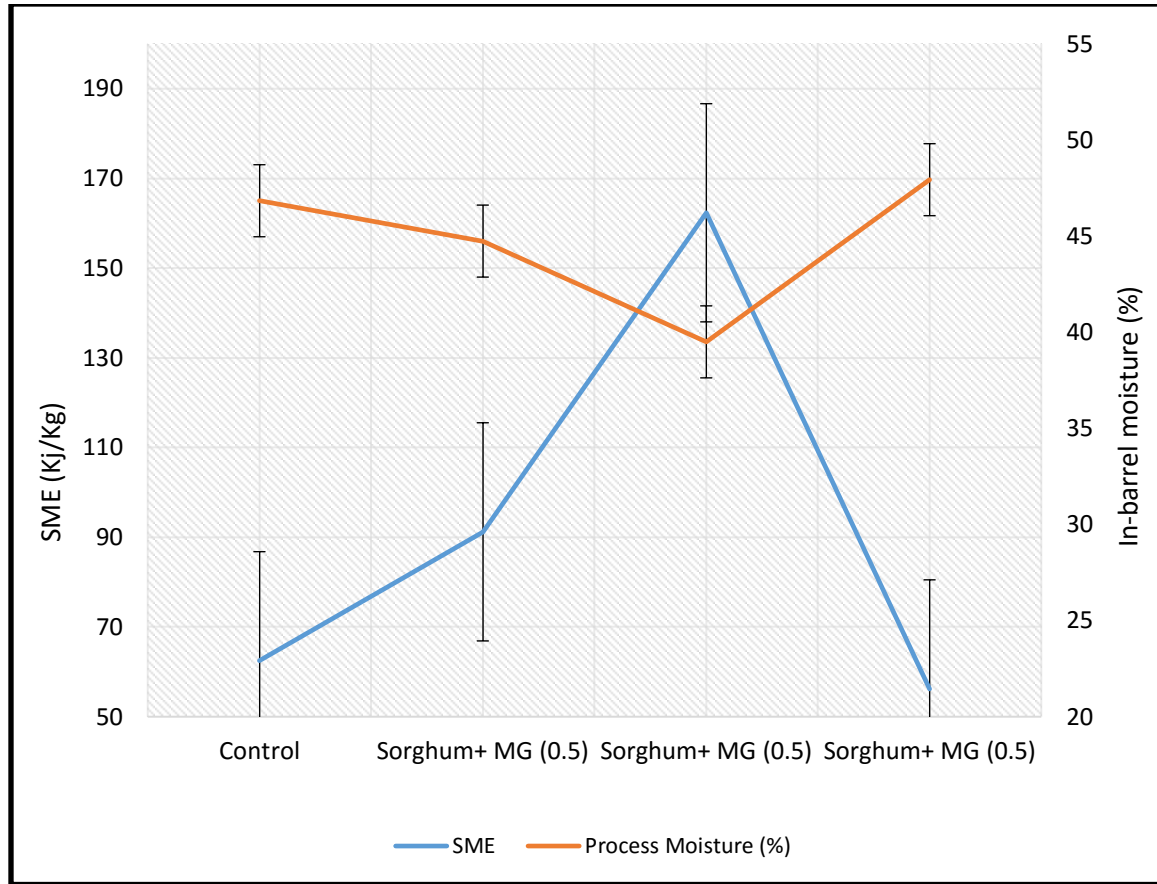


Figure A.33:Effect of in-barrel moisture content on specific mechanical energy (SME) in sorghum pasta processing.



Control- durum wheat pasta, MG- mono-glycerides

APPENDIX B -Tables

Table B.1: Moisture loss of pasta during oven drying.

Treatments	Raw blend (%)		After extrusion (%)		After Drying (%)	
	Teff	Millet	Teff	Millet	Teff	Millet
Control	30.5±0.2 ^a		27.4±0.4 ^b		12.67±0.1 ^a	
MG(0.5)	31.5±0.5 ^a	29.7±0.2 ^a	27.2±0.0 ^b	25.5±0.1 ^a	12.3±0.1 ^a	11.0±0.2 ^d
CS28AM (10)+MG(0.5)	31.3±0.1 ^a	29.7±0.7 ^a	25.7±0.1 ^a	27.7±0.8 ^b	12.1±0.3 ^a	06.0±0.1 ^b
CS28AM (10)+MG(1)	31.0±0.2 ^a	28.9±0.2 ^a	28.1±0.1 ^b	24.6±0.1 ^a	11.4±0.1 ^b	04.4±0.6 ^c
CS28AM (20)+MG(0.5)	31.0±0.1 ^a	28.6±0.6 ^a	27.4±0.1 ^b	24.6±0.2 ^a	12.5±0.0 ^a	12.8±0.1 ^a
CS28AM (20)+MG(1)	30.6±0.1 ^a	28.7±0.3 ^a	27.3±0.1 ^b	27.3±0.6 ^b	11.6±0.0 ^b	11.1±0.1 ^d
CS55AM(10)+MG(0.5)	28.9±0.3 ^b	29.5±0.6 ^a	26.5±0.1 ^b	24.7±0.6 ^a	12.7±0.0 ^a	10.0±0.1 ^d
CS55AM(10)+MG(1)	30.7±0.9 ^{ab}	29.5±0.0 ^a	26.6±0.1 ^{ab}	24.7±1.4 ^a	11.7±0.0 ^{ab}	04.7±0.2 ^c
CS55AM(20)+MG(0.5)	30.5±1.0 ^{ab}	29.4±1.8 ^a	27.2±0.1 ^b	25.5±0.9 ^a	12.2±0.1 ^a	12.1±0.1 ^{d^a}
CS55AM(20)+MG(1)	30.5±0.2 ^{ab}	27.9±0.1 ^b	27.2±0.1 ^b	25.3±0.9 ^a	11.8±0.1 ^{ab}	07.5±0.0 ^b
Coarse Grain	28.6±0.2 ^b	33.7±0.1 ^c	27.6±0.1 ^b	28.8±0.5 ^b	10.4±0.0 ^b	12.6±0.6 ^a

Legends: Control wheat- durum wheat semolina pasta, CS28AM-28% amylose corn starch, CS55AM-55% amylose corn starch, MG(0.5)- 0.5% mono-glycerides, MG(01)- 1.0% mono-glycerides.