

# Significance of tempering methods on sorghum kernel, flour, and baking properties

by

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## Abstract

*The sorghum milling industry is not completely established unlike the corn and wheat milling industries. Current sorghum milling methods may result in lower flour yield, high milling losses, flour contamination, inconsistent flour quality, and greater damaged starch content. Appropriate tempering processes could improve the sorghum milling and bread making properties. Direct and indirect methods were applied to investigate the effects of cold water, hot water, and steam tempering methods on the sorghum kernel, flour and bread making properties.*

*In this study, three sets of tempering methods were carried out: (i) cold (16 and 18% m.c at 24°C for 24 h); (ii) hot (16 and 18% m.c at 60°C for 12, 18, and 24 h); and (iii) steam (at 20 psi for 5, 10, and 15 sec). Single kernel characteristics (SKCS), abrasive hardness index (AHI), bulk, true, and tapped density, coefficient of static and rolling friction were measured for white and waxy white sorghum kernels. The cold water tempered sorghum kernels had both lower SKCS hardness and AHI than kernels treated with other tempering methods. Overall tempering condition and tempering moisture content was found to have significant effect on the physical properties i.e. bulk, tapped, and true density, and angle of repose than material properties i.e. coefficient of static and rolling friction.*

*The effects of tempering methods on the roller milling performance and flour quality were also evaluated. The developed laboratory-scale roller milling flowsheet consists of four break rolls and eight reduction rolls. Hot water tempering method (16% m.c at 60°C for 18 h) led to better separation of bran and endosperm, due to toughening of the bran, which allowed gradual scraping of the endosperm from the bran without negatively impacting flour quality characteristics i.e. particle size distribution, flour yield, protein content, ash, damaged starch content, and moisture content of the white and waxy white sorghum flours. The effects of tempering methods on the bread*

*making characteristics were also evaluated. Bread produced from the flour obtained from milling sorghum kernels tempered with cold water (18% m.c for 24 h) and hot water (16% m.c at 60°C for 18 h) displayed better bread making properties i.e. low firmness, high resilience, greater volume index, higher number of cells, and thin cell walls when compared to other tempering conditions. Hot water tempering treatment (16% m.c at 60°C for 18 h) could be a better pretreatment process for milling white and waxy white sorghum kernels without negatively impacting the flour and bread making quality characteristics.*

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## **Chapter 1 – INTRODUCTION**

The widespread awareness of celiac disease and other gluten-related disorders has been increasing recently. Celiac disease has been reported to affect approximately 1% of the population in Europe and United States (Jnawali et al., 2016). Other gluten-related disorders such as wheat allergy, non-celiac gluten sensitivity or wheat intolerance syndrome (Valenti et al., 2017) have also been gaining rapid attention. This has had a consequential effect on the global gluten-free market. Thus, the rise in production of various gluten-free products like tortillas, bread, pastas, cookies, and several other bakery products. The global gluten-free market is estimated to expand at a Compound Annual Growth Rate (CAGR) of 9.1% from 2019 to 2025 (Grand View Research, 2019).

Sorghum is a gluten-free cereal and is widely grown in several parts of the world. The United States is the leading producer of sorghum in the world for the production year of 2019-2020 (USDA, 2020). However, most of the produced sorghum has been used for ethanol production and as animal feed. Sorghum is the fifth most important cereal crop in the world. Besides being gluten-free, sorghum is drought tolerant and can adjust to varying soil salinity and high air temperatures during growth (Rai et al., 2008). The input of energy and resources required to grow sorghum has also been reported to be less when compared to other cereal crops and produces optimum grain yield (Bean, 2020). Sorghum is also nutritious due to the presence of several antioxidants, phenolic compounds, and dietary fiber (Gordon, 2001). These nutritional benefits have been positively associated with several cardiovascular diseases and cancers (Kulamarva and Raghavan, 2009).

## 1.1. Problem Statement

Though there are various benefits of sorghum, several problems have been associated with procuring good quality flour and making baked products from sorghum. Several milling techniques have been applied in the past to mill sorghum. Initial sorghum milling was hand-pounding after addition of water, and this process was mechanized later. Though this method produced coarser flour with low oil and ash, the flour extraction rates were minimal when compared to other methods and also this method was time consuming (Reichert, 1982). Most industries currently mill sorghum by using two different techniques: first abrasively debranning the kernel and then hammer milling. This technique has some drawbacks such as greater milling loss, inconsistent flour quality, lower production rate and usage of debranner is only feasible for milling small batches (Zhao, 2016; Reichert, 1982). Roller milling was also used to produce sorghum flour with higher extraction rates and has a better scope of developing this technique at an industrial scale. However, one issue reported with this type of milling was greater ash content (Kebakile et al., 2007), which could be resolved with better tempering techniques. Wet milling was also practiced on sorghum due to its similarity in structure with corn. Previous research has indicated difficulty in starch recovery due to complex starch-protein matrix and extreme friable pericarp obstructs clean recovery of starch (Watson, 1984). The other factors which make sorghum milling complicated are its extremely friable pericarp, large integral germ and varying proportions of corneous and floury endosperm (Fig 1-1).



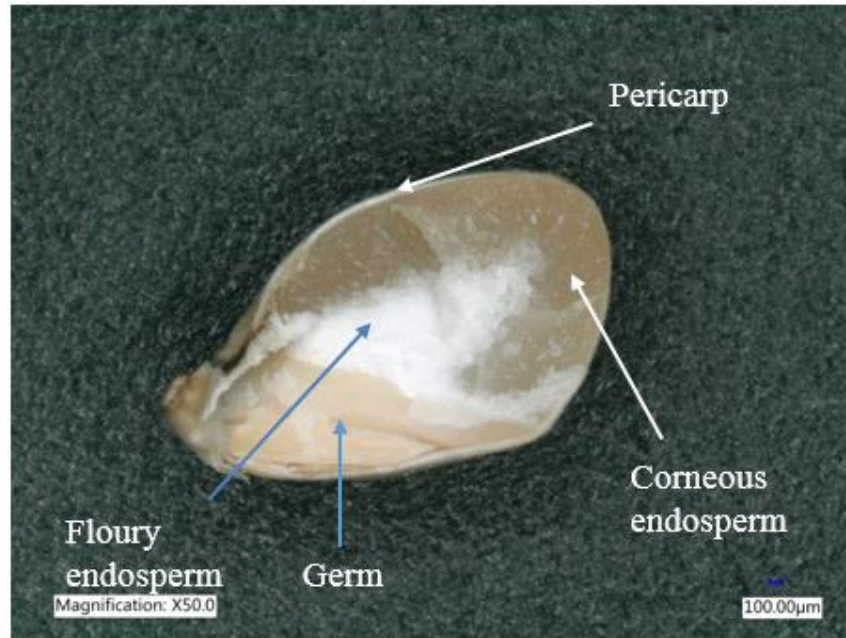


Fig 1- 1: Sorghum kernel internal structure.

Since roller milling has been proven to be better when compared to other milling techniques in terms of procuring good quality flour, proper tempering methods are imperative to improve the efficiency of roller milling. Tempering is an essential pre-milling step performed by addition of water to toughen the bran and soften the endosperm. Several tempering conditions were applied in the past to study the effective separation of pericarp and germ from the endosperm. Cecil (1992) suggested tempering the grain to 26% m.c. at 60 °C to procure maximum separation of bran, fat and fiber. However, this condition produced flour with greater moisture content which was not safe for storage. Young (1993) tempered sorghum 16% m.c. at 4 °C, which produced good flour quality, but the low temperature tempering may not be feasible. Zhao (2016) tempered red sorghum using steam, cold water and hot water and suggested that steam tempering was the most efficient tempering method to separate bran from endosperm. Fredrick (2009) evaluated the effect

of particle size of sorghum flour on gluten-free sorghum bread by using pin-milling. The pin-milling was conducted in different speeds and it was observed that the flour particle size and starch damage content of the flour was significantly affected by the extraction yield and pin-milling speed. Iva (2012) had studied the production of low-fat and low-ash content sorghum flour using different milling techniques like roller milling without decortication, hammer milling and Alpine pin milling. Through this study, it was concluded that a milling system with prebreak and reduction system produced sorghum flour with better extraction and baking qualities.

Since flour is the required final product from milling and it is the base ingredient for several food and baked products, the usage of different tempering methods/conditions have significant effects on the final product quality. Flour composition, particle size and quality are some of the factors which affect the final characteristics of the product (Lijuan et al., 2007; De La Hera et al., 2014). Proper tempering and milling conditions have direct effect on these factors. Due to the lack of gluten content in sorghum flour, making bread out of sorghum flour has also been a challenge. The combination of hydrocolloids, starch and sorghum flour helps in imitating the properties of gluten (Arendt et al., 2008). However, there is lack of knowledge on the effect of pre-processing condition (tempering method) and baking properties of gluten-free cereals like sorghum.

## **1.2. Research Overview**

Considering that the sorghum milling industry is not as established as that of corn and wheat milling industries, the purpose of this study was to develop an efficient tempering and milling technique using laboratory-scale roller mill for white and waxy white sorghum which prevents the need to develop a new milling technique particularly to mill sorghum. The effects of

tempering on kernel, milling, flour and baking properties of white and waxy white sorghum are evaluated through this research.

### **1.3. Research Hypothesis**

The hypothesis of this study is that the tempering methods impact the moisture penetration and milling behavior of sorghum kernels and thus further influence the flour and bread making properties.

### **1.4. Research Objectives**

The overall goal of this project was to determine the effects of tempering on white and waxy white kernel, milling, flour, and baking properties. The specific objectives of the study were:

1. To determine the effect of tempering methods on white and waxy white sorghum kernel properties.
2. To develop novel mill flow sheet using laboratory-scale roller mill for milling white and waxy white sorghum kernels.
3. To determine the effect of tempering methods on white and waxy white sorghum flour and bread making properties.

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## **Chapter 2 - REVIEW OF LITERATURE**

### **2.1. Celiac disease and other gluten related disorders**

Celiac Disease (CD) is an autoimmune disorder caused by the ingestion of gluten from gluten-containing grains like wheat, rye, barley, spelt and kamut (Fasano and Catassi, 2012) which can lead to inflammation of the small intestine lining causing malabsorption of essential nutrients (Volta et al., 2014). From previous analysis of patients on CD, the clinical phenotypes of CD were classified as classical, non-classical, and subclinical CD which occur to 27%, 52% and 21% of the patients diagnosed with CD (Caio et al., 2019). Classical CD patients show diarrhea, weight loss and malabsorption as symptoms, non-classical CD patients show constipation, anemia and osteoporosis as symptoms and subclinical celiac patients show no symptoms though diagnosed with CD (Caio et al., 2019). The Celiac Disease Foundation (2018) had disclosed from its recent reports that CD was widespread to about 0.4% of the population in South America, 0.5% in Africa and North America, 0.6% in Asia, and 0.8% in Europe and Oceania. The number of celiac patients is predicted to double every 15 years (Reilly, 2016). There are other disorders related to gluten, like nonceliac gluten sensitivity (NCGS)/nonceliac wheat sensitivity which is a clinical entity where the patients suffer similar symptoms to that of CD (like gastrointestinal inflammation), however, they do not get tested positive for CD (Camhi et al., 2019). It has been reported to affect around 6% of the adult population with varying proportions in different geographical regions (Camhi et al., 2019). Another related disorder is wheat allergy which is the detrimental immunologic reaction of proteins present in wheat and contemporary grains (Christensen et al., 2018). All the gluten disorders have been slowly increasing, both in terms of cases and awareness with a predicted global prevalence of around 5% (Christensen et al., 2018). These disorders have

been described to have similar symptoms and the use of gluten-free food has been the suggested scientifically unfounded remedy to all these disorders (Cabanillas et al., 2019).

## **2.2. Gluten-free market and gluten-free food products**

The widespread awareness of celiac disease and gluten related disorders has caused the global GF market to skyrocket. The global gluten-free products market size is estimated to reach USD 32.39 billion by 2025 expanding at a CAGR of 9.1% (Grand View Research, Inc., 2019). The same research forum had announced North America to be the leading gluten-free consumer market in 2018 holding a share of 53.0% of the total market. GF products have been consumed not only by celiac and gluten-sensitive patients but also health-conscious people focusing on weight maintenance, greater protein and fiber intake and diabetic patients (Morreale et al., 2018). GF flours, biscuits, cookies, bread, cakes, pastas, snacks and other innovative GF products like GF beer have been gaining popularity in the market (Masih et al., 2019). Major food companies like the Kellogg's Company, General Mills, the Kraft Heinz Company etc. have been thriving their way to compete against each other to become key players in the gluten-free market (Reilly, 2016). Around 22% of the GF food consumers in the USA are drawn to approximately 10% of the restaurants in the USA selling GF food which has been influencing other restaurants also to serve GF food (Mintel, 2016). The bakery sector has dominated the gluten-free market in 2018 (Masih et al., 2019, Grand View Research Inc., 2019).

Several researches have been conducted in the past in making gluten-free products satisfying consumer preferences. Some of them include:

*Snack Products:* Black and brown sorghum (Zelaya et al., 1999) have been cooked with alkali previously to make tortilla chips. Since both these types of sorghum contain polyphenols and



antioxidants. Alkaline-cooked whole sorghum was deep fried in fat to produce a snack product with crunchy texture. This product was developed in a research by Suhendro et al. (1998).

*Baked Products:* Cakes made with the 70:30 of sorghum or millet flour to cassava starch had better cake properties (Olatunji et al., 1992). Oyidi (1976) observed that cookies and cakes made from sorghum flour were dry, crumbled easily and had a bland flavor when compared to those made from wheat flour due to the presence of gluten that produced larger cookies and cakes with a softer and more porous structure. Sorghum breads have been explained further in the review below.

*Tortillas and Noodles:* Tortillas have been gaining attention for a long period of time. Previous research has indicated that a mixture of corn and sorghum flour has produced acceptable quality of tortillas (Choto et al., 1985). Decorticated sorghum flour, salt and water was used to make sorghum noodles in a research by Suhendro et al. (2000). This research explained the importance of amylose and amylopectin content in the sorghum flour which affected the retrogradation and gelatinization consequently influencing the noodle characteristics.

*Couscous:* Previous research has shown that hard endosperm sorghum yields more and better flour for couscous than soft endosperm sorghum (Galiba et al., 1988).

*Porridge:* Porridge is one of the staple foods in Western Africa, where sorghum flour is cooked in acid, alkali, or water to give a consistency (Smith and Frederiksen, 2000). Bangu et al. (1994) proposed that when the combination proportion of sorghum, maize and cassava was 30:40:30, it is acceptable to sensory panelists as a good quality porridge.

*Beer:* Sorghum beers are widely popular in African countries. It is a significant part of the diet of many poor people in Rural Africa (Kayodé et al., 2007).

## **2.3. Sorghum**

Sorghum lies fifth in importance after rice, maize, wheat, and barley (The United Sorghum Checkoff, 2012). According to the World Agriculture Production report released by the USDA (2020) for the year 2019/20, the world-wide production of sorghum has reduced when compared to last year and United States is the leading producer of sorghum in the world with an estimate production of 8.67 million metric tons expected for this year. After United States, Nigeria, Ethiopia, and India are the leading producers. Kansas is the leading producer of sorghum in USA. This crop is widely abundant in many parts of the world and can grow in semi-arid climates or regions which receives maximum solar energy. It is also drought tolerant and requires less water for cultivation. Besides being gluten-free, sorghum has been found to be rich in nutrients like dietary fiber, minerals, antioxidants, vitamins, and phytochemicals (Lemlioglu, 2014). Most sorghum produced in the United States has been used for ethanol production and animal feedstock, while in Africa and India, it has been used for several traditional foods and beverages. Sorghum is also a major staple food in the countries like Burkina Faso, Chad, Mali, Nigeria, and Sudan.

### **2.3.1. Sorghum Kernel Features**

The sorghum kernel is considered a naked caryopsis except for a few African varieties which contain their glumes after threshing. The kernel is spherical in shape and is available in white, red, yellow, and brown colors. The caryopsis consists of three components:

- pericarp (outer layer),
- germ (embryo and scutellum or first reserve tissue), and
- endosperm (second storage tissue).

The relative proportion of these components varies depending upon variety and environment. Generally, sorghum contains 84.2% endosperm, 9.4% germ, and 6.5% pericarp.

#### ***2.3.1.1. Pericarp***

The pericarp is the outermost layer of sorghum kernel which is generated from the ovary wall (Glennie et al., 1984) which is divided into three further layers: epicarp, mesocarp and endocarp (Earp and Rooney, 1982). The outermost layer of the pericarp is epicarp and consists of a thin layer of wax called cutin. The epicarp itself is two to three layers thick made of rectangular cells and holds the pigment material. The mesocarp layer of the pericarp lies below the epicarp and is composed of starch granules. It is usually three to four layers thick and made of small cells of starch granules. The innermost layer of the pericarp is the endocarp which is composed of cross and tube cells.

#### ***2.3.1.2. Endosperm***

Sorghum endosperm consists of an aleurone layer, peripheral, corneous and floury regions (Earp and Rooney, 1982). The aleurone layer is the outermost layer of the endosperm consisting of testa or tube cells which are arranged in a single layer of rectangular cells. This layer consists of cells with thick walls and contain large amounts of protein, ash, and oil. The peripheral layer of the endosperm consists of a dense composition of large amounts of protein and small starch granules. The corneous and floury endosperm mainly comprises of starch and protein matrix and thin cell walls rich in glucuronoarabinoxylans. In the corneous endosperm, the starch granules and protein bodies are embedded in the continuous phase of the protein matrix. The floury endosperm is right in the center of the endosperm with starch granules and protein bodies loosely packed with air pockets in the endosperm (Serna-Saldivar and Rooney, 1995).

### **2.3.1.3. Germ**

The germ or embryo consists of two major parts: the embryonic axis and the scutellum. The new plant that can be developed from a seed starts from the embryonic axis. The new plant developing from the embryonic axis divides into the radicle and plume, where the radical forms the roots and grows downward towards the soil, while the plume grows into shoots upwards. The scutellum is reserve tissue with large amounts of oil, protein, enzymes, and minerals and serves as the connection between the endosperm and germ (Serna-Saldivar, 2010; Serna-Saldivar and Rooney, 1995).

### **2.3.2. Chemical Composition of sorghum kernel**

The proximate composition of sorghum kernel widely depends on several genetic and environmental factors. The outer layer pericarp is rich in fiber. The endosperm comprises mostly of starch and protein with low amounts of fat and fiber. The germ/embryo is rich in crude protein, fat, and ash.

#### **2.3.2.1. Carbohydrates**

Non-structural (starch, sugars and fructosan) and structural carbohydrates (cellulose, hemicellulose and pectic substances) are present in sorghum. Starch is the main source of energy required for germination. Approximately 50-75% of total sorghum grain weight comprises of starch which is primarily made up of straight-linked amylose chains (glucose unit held together by  $\alpha$  (1-4) glycosidic bonds) and branched amylopectin chains (glucose units held together by  $\alpha$  (1-4) and  $\alpha$  (1-6) glycosidic bonds). Granules present in the corneous endosperm are small and angular, while those in floury endosperm are larger and round (Rooney and Pflugfelder, 1986).

The dominant and fundamental sugars present in sorghum are the monosaccharides (glucose and fructose), disaccharides (sucrose and maltose) and trisaccharides (raffinose) (Rooney and Clark, 1968).

#### **2.3.2.2. Fiber**

Most of the sorghum fiber is in the pericarp and endosperm cell walls and mainly comprises of cellulose, hemicellulose, lignin, pectin and gums. Most of the fiber present in sorghum is not soluble (86.2%) (Leder, 2004).

#### **2.3.2.3. Proteins**

Sorghum contains an approximate of 11% of protein (Serna-Saldivar and Rooney, 1995). However, this quantity depends on varying agronomic conditions like water availability, soil fertility, temperature, and environmental conditions during grain development. The endosperm, germ, and pericarp consist of approximately 80, 16, and 3% of the total protein available in sorghum, respectively (Taylor and Schussler, 1986). Sorghum endosperm contains a lot of prolamines. It particularly contains glutelins and kafirins (sorghum prolamines) that are close to 80% of the total proteins available in sorghum (Serna-Saldivar and Rooney, 1995). Kafirins are fundamentally located in spherical protein structures that are tightly packed in a protein network, embedded in a glutelin protein matrix enclosed by starch granules (Taylor and Schussler, 1986). Glutamic acid, leucine, alanine, proline and aspartic acid are the available amino acids in sorghum (Taylor and Schussler, 1986).

#### **2.3.2.4. Lipids**

The total lipid content present in sorghum also depends on a lot of external factors that has affected sorghum growth. The lipid content in sorghum varies from 2.1-6.6% (Serna-Saldivar and Rooney, 1995). The germ of the kernel contains close to 80% of the total lipid content present in sorghum. Maize and sorghum consist of similar lipid composition: linoleic (49%), oleic (31%), and palmitic (14.3%) acids (Hoseney et al., 1981).

#### **2.3.2.5. Vitamins and Minerals**

Minerals are predominantly available in the pericarp, aleurone layer and germ which are mostly lost during processing (Serna-Saldivar and Rooney, 1995). Thiamine, riboflavin, and niacin in sorghum are the most widely available B vitamins in sorghum. Other vitamins like D, E and K have also been traced in sorghum (Rooney, 1996).

#### **2.3.2.6. Tannins and phenolic acids**

All types and varieties of sorghum consist of phenolic acids which are in pericarp, testa and aleurone layer of the kernel. The most widely available phenolic acids in sorghum are: syringic, protocatechuic and caffeic (Awika and Rooney, 2004). These phenolic acids play an important role in the defense mechanism of the plant against pests and pathogens (Awika and Rooney, 2004).

### **2.3.3. Uses of Sorghum**

The presence of fermentative sugar in the stem of sweet sorghum enables it to be used for ethanol production. Sorghum has a greater proportion of starch and oil when compared to wheat. Hence, starch extracted from sorghum could be converted into syrups, modified starches, dextrose

etc. Sorghum can also be used in the preparation of beer by using it as a substitute for barley malt (Olatunji et al., 1993). Besides this, the increasing demand for gluten-free products globally has also covered the use of sorghum in making bread (Schober et al., 2005; Ari Akin et al., 2019), cookies (Chiremba et al., 2009; Rao et al., 2018), pastas (Orlandin et al., 2019) etc.

## **2.4. Sorghum Milling**

Sorghum can be milled using dry or wet milling. The main objective of dry milling is to efficiently and cleanly separate the bran, endosperm and germ while wet milling is used to procure pure starch, lipids and protein compounds from the germ and endosperm. The main factors that affect sorghum milling are kernel shape, friable pericarp and the variable proportion of corneous to floury endosperm. The endosperm of sorghum is extremely hard compared to other cereals like hard wheat, soft wheat, and barley.

Milling sorghum grain into flour is not as established as wheat or corn milling techniques. The particle size of gluten-free flours is very important as it has serious effects on dough characteristics which consequently affects the final bread quality. The milling of sorghum can be categorized as: Hand-pounding, abrasive type dehullers, roller milling, and wet milling.

### ***2.4.1. Hand-pounding***

In the primitive years of sorghum milling, the grain was laboriously and time-consumingly pounded using pestle and mortar, which was later mechanized using small grinders in several villages. In this method, sorghum kernels were first mixed with water. The wet kernels were then pounded, and the bran was removed by sifting. However, this method had most of the germ

retained in the endosperm which was converted to flour. There were relatively high losses of nutrients observed in this method (Kebakile et al., 2007).

#### ***2.4.2. Abrasive-type dehulling***

This technique employed carborundum or other abrasive surfaces mounted on a vertical or horizontal rotor to scrape or abrade the outer layers of the kernel. This technique is currently employed in most small-scale to medium-scale milling industries. The dehuller scrapes the outer surface of the kernel and the light bran and dust are removed by screening while the larger bran particles are removed by air aspiration. Dehulling is affected by the number of times the grain passed through the dehuller. Though this method was found to efficiently remove the bran and germ layers (pericarp and germ) which also reduces tannin and phytic acid contents. This technique has a few setbacks like resulting in lower milling yield and greater protein loss due to soft endosperm of tannin-containing sorghum. The decorticated material was hammer milled, separated using gravity separator and sieved to produce grits with low fat, meal and flour (Reichert et al., 1988). Flour protein content was correlated with bran removal during milling, and it was proposed that, the removal 18% of the kernel during debranning could increase the flour yield by 8-10% with a greater protein content (Rooney et al., 1972). Recent research by Iva (2011) proposed that the current decortication step for separating bran and endosperm was not economically viable.

#### ***2.4.3. Roller Milling***

Roller milling has been the principle grinding technique for wheat milling industries. Roller mills work on the principle of shear and compression caused by the corrugations on the roll surfaces. The pressure exerted on the kernel by the corrugation on the roll hold the kernel and



shears to break the kernel to smaller fragments. There are several factors which affect the working of roller mills, some of them are: the flowrate of stocks to the rolls, the differential between the fast and slow rolls, the gap between the rolls, the type and condition of the rolls etc. Sorghum milling was also developed with the introduction of roller mills used to mill wheat. Kebakile et al. (2007) used two to three pairs of roller mills to mill sorghum. These roller mills performed an efficient job in opening the kernel with bran intact using the break rolls and the reduction rolls reduced the fragments to meal or flour. Milling was particularly focused on removal of bran and germ. Removal of bran during milling was reported to increase the protein content of flour, however reducing its quality. Germ removal also reduces lysine (sorghum's primary essential amino acid), lipid (tocopherols), essential B vitamins and mineral contents. (Taylor and Schussler, 1986). A recent study by Iva (2011) to procure low fat and low ash content in sorghum flour also reported that white sorghum milled using roller mills with a prebreak and a gradual reduction system produced flour with higher extraction rate and good baking properties. Another recent research by Zhao et al. (2016) milled red sorghum using laboratory-scale roller mill after tempering with cold water, hot water and steam.

#### ***2.4.4. Pin-milling***

Frederick (2009) milled white food grade sorghum using pin-milling that produced flour with three extraction rates (60, 80 and 100%). The flour characteristics like flour composition, damaged starch, total starch content, particle size distribution and water absorption were analyzed. The baked breads were analyzed for specific volume, and crust and crumb characteristics. Flour characteristics like crude fiber content, total starch content, damaged starch and flour particle size were significantly affected by pin milling extraction rate and speed of pins' rotation. The bread

baked from 60% extraction rate of flour provided the best result in terms of specific volume and crumb firmness. This study firmly associated bread characteristics with flour properties like particle size, damaged starch content and crude fiber content.

#### ***2.4.5. Wet-milling***

Sorghum is wet-milled according to a similar procedure to that of corn (Watson, 1984) due to similar kernel structure and composition to that of corn. This process involves steeping the grain in mostly sulfur-dioxide solution under controlled processing conditions of steeping medium concentration, time and temperature. This allows the water to diffuse into the endosperm, germ and other cellular components. The issues with wet milling of corn are the difficulty in starch recovery unlike for corn due to its kernel structure. The friable pericarp of sorghum also obstructs the proper separation of starch and protein resulting in production of bran specs and discoloration in the starch (Wang et al., 2000).

### **2.5. Tempering**

Tempering is the process of conditioning the grains with water to toughen the outer bran and soften the endosperm (White, 1966). The tempering also increases the volume of the kernel which facilitates easy separation of germ, pericarp, and endosperm (Eckhoff and Paulsen, 1996). Dry milling requires addition of water in controlled amounts. The added water during tempering penetrates into the outer layers of the grain by capillary diffusion and is distributed throughout the endosperm and germ (Hsu, 1984). The amount of time allowed to penetrate the water into the kernel, the moisture content of the grain and the amount of water added to the grain are some of the factors which affect the conditioning of the grain (Bradbury et al., 1960). The larger proportion of corneous endosperm in the kernel makes the kernel absorb moisture slowly and previous

research has reported that, waxy sorghum will absorb more water when compared to non-waxy sorghum varieties (Mustafa, 1969).

The most common applications of tempering are cold water, hot water and steam. Cold water tempering is the conditioning done where the temperature of the conditioning water is at room temperature. Hard wheat is usually tempered with cold water to 15.5-17% and soft wheat is tempered to bring to final moisture content 14-15.5%, while the holding time ranges from 12-24 hours (Butcher and Stenvert, 1973; Berghofer et al., 2003). The wheat milling industry widely uses cold water tempering due to its low energy usage and cost of performance. When the temperatures used exceed 46 °C with varying holding times, it is termed as hot water tempering. According to studies by Jones (1948) and Swanson and Pence (1930), moisture penetration into the kernel is affected by longer duration of hot water tempering. Another research by Swanson et al. (1916) also indicated that tempering beyond 57 °C can potentially cause denaturation of protein but can improve the flour and milling quality. Steam tempering was reported to be more effective in transferring the heat to the kernels when compared to hot air or other radiators (Bradbury, 1960). The application of steam provides a combined effect of heat and moisture at the same time, the target moisture content is achieved at a faster rate when compared to the other tempering conditions (Zhao, 2016). Steam tempered wheat was reported to produce higher flour yield, coarser bran extraction and required lesser power consumption than hot water tempering methods (Schafer, 1951).

### ***2.5.1. Sorghum Tempering***

Several researches were conducted in the past to improve dry milling of sorghum by employing tempering techniques. The pericarp structure of sorghum and presence of hydrophobic

kafirin proteins make it difficult to absorb water during tempering (Munck, 1995). White sorghum was tempered at 21, 48 and 66 °C and dehulled before extracting flour (Wu, 1971). Wu (1971) proposed to increase the kernel moisture content to 17% at 21 °C and holding the tempered kernels for 1.5 h before milling was the most optimum tempering condition. It was also proposed in the same study that the flour yield, fine grits, percent fiber content and protein content decreased with increasing tempering temperature, moisture content, and holding time. Sorghum grain was conditioned for 8 h to bring to final moisture content of 17% and concluded that bran and fine grit yield increased with increasing moisture content (Abdelrahman et al., 1981). Cecil (1992) described in his study how hot water tempering at 60 °C for 6 h bringing to a final moisture content of 26% improved degermination and bran separation. However, the flour extraction rate and keeping quality of the flour reduced due to the high moisture content of flour. Gomez (1993) suggested from his study that conditioning sorghum to 16% final moisture content at 4 °C for 24 h improved flour extraction with flour at a microbiologically safe level. The drawback with this technique was the lack of proper tempering technology that industry could adapt as that of wheat and corn milling. Iva (2011) also tempered white food grade sorghum to final moisture contents: 14.5, 16.5 and 17.5% for 3, 5 and 7 h before roller milling and observed that there was no significant difference in flour yield between the different tempering moisture contents. The bran yield was observed to increase significantly with increasing tempering moisture content. Zhao (2016) studied the effects of cold water, hot water and steam tempering conditions on the roller milling and flour quality of red sorghum. This study proposed that bran yield increased significantly with steam duration and had resulted in low damaged starch content (3.6 - 4.2 (%dry basis)) when compared to cold water treatment. Zhao (2016) also proposed that the application of

steam at high temperature and pressure led to better separation of bran and endosperm without impacting the flour quality.

## **2.6. Gluten-free bread**

The FDA (2014) had introduced the following guidelines for food to be labelled as GF:

- If the food is produced from cereals which are completely devoid of gluten (e.g., sorghum)
- If the food is not produced from any cereal consisting gluten (e.g., wheat, barley, rye, spelt)
- If the food does not contain any ingredient that has been processed to remove gluten but obtained from a gluten-containing cereal (e.g., wheat starch).
- If the food contains any ingredient that has been derived from a gluten-containing cereal and processed to remove gluten but is present more than 20 ppm.
- If there is an unavoidable inclusion of gluten in the food greater than 20 ppm.

Gluten is protein network that is formed when wheat flour is hydrated and, glutenin and gliadin proteins bind together. With continued mixing, the protein chains increase in number and elongation which improves the webbing that expedites the ability of the dough to stretch (elasticity) and the ability to retain shape (extensibility). The elasticity and extensibility traps gases during fermentation and allows expansion to produce a fully developed baked product. The lack of gluten in gluten-free flours lack the network forming capacity required to form an enclosed trap for gases during fermentation and rising which produces good quality bread. The absence of gluten causes the hydration of GF flours to form a “cake-like batter” rather than a “viscoelastic” dough helpful for bread formation (Cauvin, 2015). This has led to the use of substitutes of gluten which perform in a similar way to gluten. Previous research has indicated that GF bread lack quality,

good mouthfeel in terms of texture, good crust and crumb grain characteristics and accelerated staling (Gallagher et al., 2003).

A GF bread consists of the following major ingredients to bring up the quality close to a wheat-pan bread: GF flour, starches, hydrocolloids, animal proteins and vegetable proteins (Arin Akin, 2017). The following are the functions of different ingredients used to make GF bread (Carson, 2017; Zannini et al., 2012; Houben et al., 2012):

### ***2.6.1. GF flours***

GF flours are procured from GF cereals like maize, rice, millet and sorghum; legumes like chickpea, lentils; flours from nuts like chestnut and tiger nut (Arin Akin, 2017; Puig, 2015; Vivas, 2013). GF flour provides the required nutrition level, protein content and essential amino acids required to build the structure of the bread. Legume flours like chickpea flour, pea isolate and soya flour have been used to make gluten-free breads where chickpea bread showed softer crumb with better physico-chemical and sensory characteristics (Minarro et al., 2012). Another research previously conducted indicated that if 25% quinoa flour was incorporated with rice flour, potato starch and buckwheat flour, the technological properties of bread improved due to improved viscosity of the batter (Tarkut et al., 2016). Rice flour was also indicated to be widely consumed and popular among wheat-intolerant or celiac patients (Torbica et al., 2010). Marco and Rosell (2008) stated that rice flour has been popularly used in making gluten-free foods due to its neutral flavor, low sodium levels, better digestibility rate and it possessed proteins that are not very likely to cause allergy.

### **2.6.2. Starches**

Starches improve the extensibility and viscoelastic of the dough facilitating entrapment of gases during fermentation and increase hydration capacity of the dough. The addition of starch causes gelatinization to occur at a faster rate and helps in the development of a crumb network that traps gasses preventing the crust from collapsing (Taylor et al., 2006). Wheat starch, corn starch and potato starch have been widely used in making sorghum bread. Onyango et al. (2011) in his research pointed out that when sorghum bread was prepared from cassava starch with 50%, displayed better crumb properties like firmness, springiness or chewiness. Previous research by Schober et al. (2007) also indicated that lower pasting temperature of potato starch produced better sorghum bread than from maize starch.

### **2.6.3. Gums and hydrocolloids**

Gums and hydrocolloids function as a thickening agent to aid swelling, stabilization, and gelatinization. Hydrocolloids are used to improve the texture and appearance of gluten-free baked products (Akin, 2017). Xanthan gum has been used in several previous researches on gluten-free bread preparation due to its ability to maintain a stable viscosity nature with varying temperatures (Akin, 2017). Hydroxypropyl-Methyl-Cellulose (HPMC) has been discussed to increase specific loaf volume, improve gas retention capacity and crust and crumb grain texture. Locust bean gum has also been previously used in researches where it facilitated in increasing the height of gluten-free bread loaves and lag staling period.

### **2.6.4. Proteins**

Proteins provide firmness, stability and structure to the bread loaf. Egg proteins contain the strong ability to form viscoelastic films which helps in containing the gas during fermentation

(Akin, 2017). This vividly helps in producing good crumb grain texture and maintaining bread loaf volume (Onyango et al., 2011).

#### ***2.6.5. Fat, oil and emulsifiers***

Lipids used for bread making primarily act as conditioners to make the final loaf soft. They also act as a lubricant which facilitate efficient mixing of ingredients. Fats also play an important role in prolonging the shelf-life of the final product. They also help in incorporating air and stabilizing the dough.

#### ***2.6.6. Sorghum Bread***

Several previous researches have indicated that sorghum bread has been challenging to produce. Sorghum flour has always been accompanied with the addition of other starches like potato, cassava or maize starch for better dough properties. From a research by Schober et al. (2007), the use of 2% hydroxypropyl methylcellulose improved the quality of the sorghum bread with the formulation 70:30 of sorghum flour: potato starch and 105% of water. The physical appearance of the crumb was further improved by sourdough fermentation of sorghum flour in the same research. Several other studies have also been conducted by making bread from composite flour which consists of wheat flour. However, such breads are not completely gluten-free and are not advisable to be consumed by gluten-tolerant patients. Another research conducted by (Schober et al., 2005) indicated that sorghum bread made with a combination of sorghum flour and corn starch show larger volumes with the batter having a lower viscosity. The same research also concluded that the use of xanthan gum and skim milk powder produces negative effects on the crumb structure. Sorghum bread had also been previously made using sourdough for better bread qualities (Schober et al., 2007; Ogunsakin et al., 2015). Sorghum bread has been baked using both



yeast fermentation (Fort, 2016) and using chemical leavening agents (Akin, 2017). Most sorghum breads made in the past were a fusion of wheat flours and sorghum flour. However, these breads made were not considered as safe for celiac patients.

The popularity of sorghum has been increasing with years and has had several advantages. Though there have been several researches conducted in the past, there has not been any continuous study done on linking sorghum tempering with milling, flour composition and bread study on food grade white and waxy white sorghum.

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## Chapter 3 – SIGNIFICANCE OF TEMPERING PROCESS ON SORGHUM KERNEL PROPERTIES

### 3.1. Abstract

*Physical and mechanical properties of white and waxy white sorghum tempered with cold water to bring to final m.c. 16% (w.b) and 18% (w.b), hot water for 12, 18 and 24h to bring to final m.c. 16% (w.b) and 18% (w.b) and steam at 20 psi for 5, 10 and 15s were evaluated. The bulk and tapped density were found to increase with increasing moisture content when treated with cold water, hot water or steam. Abrasive hardness index did not display any noticeable trend with increasing moisture content or tempering method. When untempered white and waxy white sorghum was tempered using steam at 20psi for 15s, the kernel properties like hardness index decreased from  $(69.42 \pm 1.83)N$  to  $(56.43 \pm 0.34)N$  and,  $(76.47 \pm 0.73)N$  to  $(62.08 \pm 1.35)N$  for white and waxy white sorghum kernels, respectively; mean kernel diameter increased from  $(2.95 \pm 0.01)mm$  to  $(3.45 \pm 0.06)mm$  and,  $(3.59 \pm 0.01)mm$  to  $(4.16 \pm 0.01)mm$  for white and waxy white sorghum kernels, respectively; mean kernel weight increased from  $(24.29 \pm 0.27)mg$  to  $(26.10 \pm 0.61)mg$  and  $(36.07 \pm 1.12)mg$  to  $(39.99 \pm 0.52)mg$  for white and waxy white sorghum kernels, respectively. Steam tempered sorghum also had higher coefficient of rolling and static friction and angle of repose when compared to sorghum conditioned with other tempering methods. Steam tempered sorghum at 20 psi for 15s exhibited the most desired kernel properties when compared to all ten treatments. However, kernels tempered using this treatment had higher moisture contents around 22% (w.b.) which is usually unfavorable for long-term storage.*

**Keywords:** *white sorghum, waxy white sorghum, tempering, physical properties, mechanical properties*

### 3.2. Introduction

The information of engineering properties, especially physical, material, and mechanical properties of grains are very much essential for the process engineers in designing the grain handling, storage, and processing equipment as well as operations. Data on the engineering properties of white and waxy white sorghum as an effect of tempering process conditions are essential for designing the handling and processing operations. To our knowledge this will be the first research to document the engineering properties of white and waxy white sorghum.

Sorghum is the fifth most cultivated cereal crop in the world. The leading producers of sorghum in 2013 were United States, Nigeria, Mexico, India, and Sudan with total production of more than 61 million metric tons (Mundia et al., 2019). According to Mundia et al. (2019), majority of sorghum production till 2018 was in Africa (41%), followed by the North America (38%) and Asia (18%). Sorghum is one of the top 20 commodities produced in USA (FAO Report, 2017) and its gross production value has increased from \$1,000 K to \$1,800 K from 2006 to 2016 (FAO Report, 2016) with most of the crop being used for ethanol production and animal feed (Wang et al., 2000).

Sorghum is known to be drought tolerant and adjustable to soil salinity and high air temperatures (ICRISAT/FAO, 1996; Rai et al., 2008). Sorghum also produces satisfactory grain yield in the absence of the optimum factors that are necessary for growth (Ragaei et al., 2006). In addition, sorghum is nutritious due to the presence of phenolic compounds which occur in the form of phenolic acids, flavonoids, and condensed tannins (Serna-Saldivar and Rooney, 1995) which have antioxidant properties that can help to protect from degenerative diseases like heart disease and cancer (Rhodes and Price, 1997). However, sorghum food digestibility is still an issue due to several factors like changes in its starch and proteins during cooking (Duodo et al., 2003 and Wu

et al., 2007). The slow digestion of sorghum starch and proteins indicates that sorghum may have low glycemic index which would be helpful for diabetic patients (Ratnavathi et al., 2016). According to Jnawali et al. (2016) Celiac disease which is a long-term intolerance to gluten ingestion occurs to approximately 1% of the population in Europe and in the US and the only therapy for people suffering from Celiac disease till present date is to strictly adhere to a gluten-free diet (Rubio-Tapia et al., 2013).

Due to the continued rise in the number of people diagnosed with Celiac disease and other disabilities to gluten tolerance, the gluten-free food market has been statistically found to increase from \$700 million in 2006 to \$5 billion by 2015 (Gallagher et al., 2004; Bogue and Sorenson, 2008). Sorghum being gluten free serves best to the expanding gluten-free market and also farmers due to its easier cultivation (Taylor et al., 2006).

Despite several advantages of this crop, the sorghum food industry remains less recognized due to inadequate milling techniques and various problems associated with the milling (Kebakile, 2008). Although the sorghum milling industry has been evolving in the years, it is not very established when compared to maize, wheat, and rice milling industries (Beta et al., 2000). Traditionally, sorghum was hand-pounded to produce flour, however, this method produced coarser meals with bran contamination (Kebakile, 2007). Commercial small-scale and medium-scale sorghum milling industries involves two processing conditions: (i) debranning the sorghum kernels and; (ii) reducing the endosperm into meal or flour (Taylor, 2003). In Africa, sorghum is first decorticated or debranned by abrasion followed by hammer milling of endosperm material to produce flour (Taylor, 2003). Roller milling was introduced to mill sorghum several years after the use of abrasive decortication-hammer milling (ADHM).

Research conducted by Kebakile et al. (2008) used two pairs of roller mills to mill sorghum where the first pair of rolls was used to break the kernel by retaining the bran and the second pair of rolls were used to separate the bran from the meal and produces flour. The authors have also compared its performance with ADHM and concluded that roller milling produced finer flour with greater ash and oil contents and, gave higher extraction rate compared to ADHM (Kebakile et al., 2008). Research by Anderson et al. (1969) also obtained similar results when sorghum was milled using roller mills. Since sorghum has a similar kernel structure to that of corn, wet milling of sorghum was also performed in the early 1970's (Adeyemi, 1983), however, obtaining starch from sorghum was more difficult when compared to corn due to its small pearl like structure (Watson, 1984). The fragile pericarp of sorghum when compared to corn hindered the separation of starch and protein and interfered in the production of bright colored flour by dispersing as speckles of bran (pericarp) in the flour (Wang et al., 2000). The greater proportion of corneous endosperm in sorghum also made it difficult to extract starch using wet milling (Wang et al., 2000).

Proper tempering treatment is necessary prior to sorghum milling to obtain efficient milling yields with less contamination of bran. Studying the physical and mechanical properties of treated grain gives an insight on the effects of tempering method on the grain handling, storage, and milling (Baryeh, 2002). Few researches have been conducted in the past on tempering sorghum where sorghum was treated with steam before producing flaked sorghum (McDonough et al., 1996) and with water at room temperature (25°C) before popping (Gaul and Rayas-Duarte, 2008). Zhao et al. (2016) had studied the effects of cold water, hot water, and steam tempering on red sorghum kernel physical and mechanical properties. In their research, it was observed that the hot water tempering was more effective than cold water tempering in improving the bran resistance

and steam tempering was more effective in producing brighter flour with lesser bran contamination than the other two tempering conditions (Zhao et al., 2016).

As a preliminary step prior to the development of milling process, this study evaluated the effects of different tempering conditions (cold, hot, and steam) on the white and waxy white sorghum kernel characteristics.

### 3.3. Materials and methods

#### 3.3.1. White and Waxy White Sorghum Sample Preparation

White and waxy white sorghum kernels were chosen for this study. 45.4 kg of each variety of sorghum free from foreign matter such as dust, dirt, stones and chaff were procured from Nu Life Market, Scott City, KS. The initial moisture content (m.c.) of white and waxy white sorghum kernels was measured using ASABE Standard S352.2 method (ASABE, 2012):

$$\text{moisture content (wet basis)} = \frac{m_w}{(m_w + m_d)} \times 100 \quad (1)$$

Where,  $m_w$  = the mass of water removed (g)

$m_d$  = the mass of sample after drying at 130° C for 18h (g)

The initial moisture content (wet basis (w.b.)) of white and waxy white sorghum were found to be  $13.46 \pm 0.08$  % and  $13.29 \pm 0.16$  %, respectively

*Cold water tempering:* White and waxy white sorghum samples were tempered with predetermined amount of cold distilled water (water available at room temperature) (required water quantity was measured using eq 2 (Balasubramanian, 2001)). The tempering process was carried out in a rotating drum to bring them to the final moisture contents of 16 and 18% (w.b).

These moisture contents used in this study were chosen based on the previous research conducted by Zhao et al. (2016) on red sorghum.

$$Q = \frac{W(M_f - M_i)}{(100 - M_i)} \quad (2)$$

Where, Q = amount of distilled water required for tempering (ml)

W = the weight of sample required to be tempered (Kg)

M<sub>f</sub> = moisture content (% w.b) to which the grain must be tempered

M<sub>i</sub> = initial moisture content (% w.b) of sample

The cold water tempered samples were air tightly packed in zip lock bags. The zip lock bags containing the cold water tempered samples were held at room temperature for 24 h for uniform distribution of moisture throughout the samples.

*Hot water tempering:* The samples were treated with the pre-calculated amount of cold distilled water (based on eq 2) in the rotating tempering drums to bring them to final m.c. of 16 and 18% (w.b.). The treated samples were then filled into glass beakers and sealed with aluminum foil to prevent water evaporation. The beakers containing the treated samples were placed in hot water bath at 60°C for 12, 18, and 24 h (Cecil, 1992). The glass beakers were shaken intermittently for every 30 min for moisture equilibration. After conditioning the samples for the respective time periods (12, 18, and 24 h), sample were cooled at room temperature prior to the evaluation of the kernel properties.

*Steam Tempering:* The samples were tempered with steam at 20 psi for 5, 10, and 15 s in a hollow container with a screw passing horizontally through the inside of the container. The screw was used to mix the sorghum samples as steam passed through the inlet which allows the uniform

distribution of steam throughout the sample. After the samples were steam treated, the samples were spread on flat trays for about 8 h to evenly dry the samples, which was achieved by ambient air drying. The samples were then transferred to ziplock bags and stored in refrigerator at 5° C until the experimental measurements.

### **3.3.2. Bulk Density**

The bulk density of the tempered and untempered sorghum samples was measured using Winchester cup arrangement (Seedburo Equipment Co., Des Plaines, IL). The samples were allowed to fall freely into a cup of volume 1 pint (1 pint =  $4.732 \times 10^{-4} \text{ m}^3$ ) from a funnel set at a height of 10 cm from the mouth of the cup. The cup was allowed to completely fill, until excess sample began to overflow. The excess grain was removed by passing a scraper in a zigzag motion. The bulk density of the samples was then calculated from the weight of the sample and volume of samples in the cup of known volume.

### **3.3.3. Tapped density**

Tapped density measures the reduction in grain volume that occurs during handling and transportation (Bian et al., 2015). Tapped density of both tempered and untempered sorghum samples was measured using Autotap Density Analyzer (Quantachrome Instruments, Boynton Beach, Fla).  $100 \pm 1$  g of grain sample was filled into the cylinder of known volume, and the cylinder was then tapped using Autotap Density Analyzer for 750 times. The tapped density was calculated as the ratio of mass of sample taken to the final volume after tapping.

#### **3.3.4. True Density**

A gas pycnometer (AccuPync II 1340, Micromeritics, Norcross, Ga.) was used to measure the true density of both tempered and untempered sorghum samples. Helium gas was diffused into the chamber to fill the entire volume of the chamber. The volume occupied by the sample and the helium gas in the closed chamber was measured by the pycnometer. The true density of the sample was then determined as the ratio of mass of sample taken to the volume of sample occupied by the solid particles in the chamber.

#### **3.3.5. Porosity**

The porosity of the sample takes into account the amount of void spaces present in the test sample for a given moisture content (Masane et al., 2016). The porosity of tempered and untempered white and waxy white sorghum kernels was evaluated using the bulk and true density values of the kernels. Eqn (3) taken from Mohsenin (1986) and Sacilik et al. (2003) was used to calculate the porosity values.

$$Porosity (\%) = \left(1 - \frac{Bulk\ Density}{True\ Density}\right) * 100 \quad (3)$$

#### **3.3.6. Hardness Indices and Kernel Properties**

The United States Department of Agriculture – Agriculture Research Service (USDA-ARS) at Manhattan, Kansas developed the Single Kernel Characterization System and has been commercialized by Perten Instruments, Sweden. It is the most advanced system for evaluating the mean kernel weight, diameter, hardness and moisture of usually 300 individual wheat kernels



(Martin et al., 1993; Martin and Steele; 1996, Osborne et al., 1997). Since SKCS was initially developed for analyzing wheat, a study was conducted by Pedersen et al. (1996) where the SKCS prototype instrument was used to evaluate the hardness of 64 genotypes of sorghum and compared with the seed vitreosity. Another study by Bean et al. (2006) to evaluate the usage of SKCS for sorghum samples and compared with the traditional methods for procuring hardness index, weight and diameter of sorghum. This study reported that SKCS measurements and measurements from traditional methods were highly correlated. This SKCS system was used in the present study to evaluate the hardness index, mean kernel weight, diameter, and moisture content of sorghum samples. The SKCS properties were measured by crushing 300 kernels between an indented rotating wheel and a smooth crescent surface (Martin et al., 1993).

### ***3.3.7. Abrasive Hardness Index***

The amount of force required to abrade against the surface of the grain kernel is calculated as the abrasive hardness index. Abrasive hardness index (AHI) was determined using a TADD (Venebles Machine Works, Saskatoon, Canada). The working principle of the instrument was explained by Oomah et al. (1981) where  $10 \pm 0.1$  g of sample placed in cups was allowed to abrade against a 80-grit abrasive disk and the time required to reduce the weight of sample by 1% was calculated as AHI (Mwasaru et al., 1988).

### ***3.3.8. Angle of Repose***

The angle made by the slope of the grain with the horizontal when the grain is allowed to freely fall on a flat base is defined as the angle of repose. In this study, 450 g of sample was allowed to freely fall onto a flat surface from a funnel held height at a height of 10 cm above the horizontal

flat surface. The diameter (d) and height (h) of thus formed grain pile was measured and the angle of repose ( $\Theta$ ) was calculated using equation 4.

$$\theta = \tan^{-1}\left(\frac{2h}{d}\right) \quad (4)$$

### ***3.3.9. Coefficient of Static Friction***

The coefficient of static friction was measured for tempered and untempered white and waxy white sorghum samples against galvanized steel using the equipment and procedure described by Subramanian and Viswanathan (2007) and Patwa et al. (2014). The apparatus consisted of a frictionless pulley fitted on a wooden board. A two-sided open cylinder was attached to a tensionless string passing through the pulley to a loading pan. The hollow cylinder resting on the plate was filled with 150 g of sample and weights were added to the loading pan till the cylinder containing the sample started sliding along the plate. The sample acted like the normal force exerting perpendicularly downwards on the plate and the weights acted as the frictional force required to move the quantity of sample in the cylindrical container. Coefficient of static friction ( $\mu_s$ ) is evaluated as the ratio of frictional force (F) to the normal force (N) as shown in equation 5.

$$\mu_s = \frac{F}{N} \quad (5)$$

### ***3.3.10. Coefficient of Rolling Friction***

150 g of was placed in a two-sided open cylinder resting on a horizontal galvanized steel plate fitted on a wooden board. The cylinder was removed to allow the sample to form a stationary cone on the plate. A magnetic compass was placed on the horizontal plate and calibrated to 0° or no inclination. The plate attached to the board was tilted on one side by rotating the screw on the

opposite side of the board. The coefficient of rolling friction measures the amount of force required to move a body of fixed weight against a surface. The angle ( $\alpha$ ) at which the sample begins to slide down the plate was measured to calculate the coefficient of rolling friction ( $r$ ) using equation 6.

$$\mu_r = \tan(\alpha) \quad (6)$$

### ***3.3.11. Experimental Plan and Statistical Analysis***

The study was a completely randomized design with 11 treatments (cold water (final m.c 16% and 18% w.b.), hot water (final m.c 16% and 18% w.b. for 12, 18, and 24h for each final m.c) and steam for 5, 10 and 15s) each for white and waxy white sorghum kernels.

The density (bulk, tapped, and true) and angle of repose measurements were conducted in triplicates. The coefficients of friction tests were performed 5 times for each sample. The abrasive hardness index and SKCS measurements were carried out with a fixed quantity of sample. The results were analyzed for statistical significance using the PROC GLM (general linear models) procedure in SAS (Statistical Analysis System) (ver. 9.3, SAS Institute, Inc., Cary, N.C.). Based on ANOVA, significant differences ( $\alpha = 0.05$ ) among the treatments and also between the two sorghum varieties were compared. The differences were recognized significant at  $p \leq 0.05$ .

## **3.4. Results and discussion**

### ***3.4.1. Bulk Density***

The bulk density for white sorghum kernels and waxy white sorghum kernels decreased in the range of about 10 and 18 kg m<sup>-3</sup>, respectively (Table 3-1). Similar results were reported for wheat (Patwa et al., 2014), sweet corn seed (Coşkun et al., 2006), minor millet grains

(Subramanian and Viswanathan, 2007), hemp seed (Sacilik et al., 2003), fenugreek (Altuntaş et al., 2005), and white and black beans (Senthilkumar et al., 2018). This is attributed to the fact that the penetration of water into the kernels increased the weight of the kernels at a slower rate when compared to the volumetric expansion of the kernel (Baryeh, 2002; Pradhan et al., 2008; Solomon and Zewdu, 2009). The addition of moisture to the kernels during tempering had reduced the flowability of the kernels. This could possibly cause the kernels to have greater amount void spaces between them which increased the volume and consequently reduced the bulk density. Irrespective of the tempering condition the bulk density values of the white sorghum kernels were significantly higher than that of waxy white sorghum kernels (Table 3-1).

Acar et al. (2019), found that the mean kernel size and mean kernel weight of hard red winter wheat showed a positive linear relationship with each other. This corroborates with the trend obtained in the bulk density between white and waxy white sorghum. Waxy white sorghum had greater mean kernel weight than white sorghum due to greater mean kernel size (Table 3-2). As mentioned above, the penetration of water during each tempering treatment had significantly influenced the volumetric expansion of the kernel when compared to its weight. Thus, irrespective of the tempering method, the volumetric expansion of waxy white sorghum kernels are significantly higher than that of white sorghum kernels and this reflected in their bulk density values (Table 3-1). The inherent greater mean kernel size of untempered waxy white sorghum kernels when compared to untempered white sorghum kernel (Table 3-2) also provides lower surface area available for water to be absorbed completely due to which a decrement in the bulk density of waxy white sorghum has been observed after tempering. There is no significant difference in between the bulk density values of cold water and hot water tempered sorghum kernels, whereas the bulk density values of steam tempered sorghum kernels are much lower than

the other two treatments (Table 3-1). This could be due the increment in moisture content of sorghum kernels during steam tempering process (Table 3-1.). During the steam tempering process, the kernels were exposed to steam at 20 psi for 5, 10, and 15 s and unlike other treatments, there was no set target of final moisture content. For the respective tempering moisture contents, the bulk density values of hot water tempered sorghum kernels didn't change with increment in tempering times (Table 3-1). This could be due to the fact that, the length of tempering time didn't significantly increase the volume/size of the sorghum kernels. Whereas, with the increment in steam tempering times

### ***3.4.2. Tapped Density***

Table 3-1 shows that there was no significant difference ( $p < 0.05$ ) in the tapped density of cold water tempered and untempered kernels. Upon comparing the impact of cold water tempering on the tapped density of white and waxy white sorghum, it can be seen that both kernels behaved differently to the tempering process. Increase in moisture content from 16 -18% (w.b) decreased the tapped density of waxy white sorghum from 819.78 to 793.88  $\text{kg/m}^3$  whereas tempering conditions had no significant influence on white sorghum. In case of hot water tempering, both types of sorghum showed a decreasing trend in tapped density with increase in moisture content. Steam tempering had significantly decreased the tapped density of the kernels. The density decreased from 862.13 to 781.40  $\text{kg/m}^3$  and 826.47 to 769.39  $\text{kg/m}^3$ , respectively for white and waxy white kernels treated with steam for 15s at 20psi. Additionally, the moisture content of steam tempered sorghum was comparatively higher with respect to hot, cold, and untempered kernels (Table. 3-1.). For instance, the initial moisture content of white and waxy white kernels was 13.46 and 13.29 % (w.b), respectively. Steam tempering (20psi) resulted in a final moisture content of

22.26 and 22.7% (w.b), respectively for white and waxy white sorghum kernels. Patwa et al. (2014) reported a similar negative relationship between moisture content and tapped density for wheat kernels. The authors infer that there is an increase in mass of kernels with addition of moisture. Moreover, tapping motion during the analysis repacks the kernels and thereby decreases its volume. As the decrease in volume is relatively less compared to the increase in mass, tapped density of the kernels were reduced. The drastic increase in the moisture content of untempered white and waxy white sorghum kernels when steam tempered at 20 psi for 15s could have possibly reduced the flowability of the kernels which had left several void spaces between kernels even after tapping due to which a greater volume was occupied by the total amount of grain taken consequently reducing the tapped density of the kernels from this tempered condition. Thus, further studying the effect of tempering on the surface characteristics of the tempered grain could possibly help to further understand the variation in tapped density values.

Tempering time had not influenced the tapped density of kernels in case of hot water tempering, whereas on tempering with steam increased time adversely affected the density. On comparing the density of white sorghum kernels with corresponding waxy white sorghum, it can be seen that the mean values are higher for former under all conditions. This is attributed to the higher mean kernel weight and diameter of untempered waxy white sorghum with respect to the white sorghum.

### ***3.4.3. True Density***

Untreated and steam treated white and waxy white sorghum exhibited the highest and lowest true density values, respectively when compared to kernels tempered with cold water and hot water (Table 3-1). Untempered grains containing less water possessed greater mass per unit

volume when compared to tempered kernels. Both white and waxy white sorghum showed a decreasing trend in true density with increasing moisture content when tempered with cold water and hot water. With the subsequent increase in moisture content of white sorghum as steam tempering time increased from 5, 10 and 15s, the true density consequently decreased to 1351.07 kg/m<sup>3</sup>, 1337.47 kg/m<sup>3</sup>, and 1325.13 kg/m<sup>3</sup> respectively. Similar positive trend was observed in waxy white sorghum with increasing steam treatment time. The results obtained from this density study agreed with results obtained from white and black beans (Senthilumar et al., 2018), coriander seeds (Coşkuner and Karababa, 2007), soybean (Shirkole et al., 2011) and millet (Baryeh, 2000). True density of white and waxy white sorghum exhibited no distinct trend with increasing heat treatment time. Untreated white sorghum displayed significantly higher true density than untreated waxy white sorghum. According to Shirkole et al. (2011) and Patwa et al. (2014), the increment in kernel volume after tempering impacted the true density of the kernel more than the increment in mass associated. Waxy white sorghum displayed greater mean kernel diameter than white sorghum (Table 3-2). Thus, waxy white sorghum could possibly expand more after tempering, leading to lower true density than the other.

#### ***3.4.4. Porosity***

From Table 3-2, it was observed that porosity values were the highest for white and waxy white sorghum kernels which were steam tempered for 15s when compared to the sorghum kernels produced from all the other tempering conditions. The subsequent increase in porosity values from 41.28% to 44.74% for white sorghum and 42.25% to 44.54% for waxy white sorghum was due to the increase in moisture content of untempered kernels (13.46% (w.b.) for white sorghum and 13.29% (w.b) for waxy white sorghum) when steam tempered at 20 psi for 15s (22.26% (w.b.) for

white sorghum and 22.70% (w.b.) for waxy white sorghum). Similar trend of increase in porosity values with increasing moisture content was observed for tender sorghum grains (Masane et al., 2016; Mwithiga and Sifuna, 2006; Sacilink et al., 2002), faba beans (Altuntaş and Yıldız, 2007) and red kidney beans (Eşref and Halil, 2007). A specific trend with increasing tempering moisture content when conditioned with cold water and hot water was not observed for both white and waxy white sorghum. There are previous which have indicated similar absence of trend in porosity values of apricot pit and its kernel (Gezer et al., 2006) and cotton seeds (Özarslan, 2002) with increasing moisture content.



Table 3- 1. Moisture content and Density (Bulk, Tapped and True) of white and waxy white sorghum for different tempering treatments <sup>[a]</sup>

| Treatment        | Moisture content (% w.b.) |               | Bulk Density (kg/m <sup>3</sup> ) |                     | Tapped Density (kg/m <sup>3</sup> ) |                    | True Density (kg/m <sup>3</sup> ) |                     |
|------------------|---------------------------|---------------|-----------------------------------|---------------------|-------------------------------------|--------------------|-----------------------------------|---------------------|
|                  | White                     | Waxy White    | White                             | Waxy White          | White                               | Waxy White         | White                             | Waxy White          |
|                  | Sorghum Grain             | Sorghum Grain | Sorghum Grain                     | Sorghum Grain       | Sorghum Grain                       | Sorghum Grain      | Sorghum Grain                     | Sorghum Grain       |
| Untempered Grain | 13.46 (0.08)              | 13.29 (0.16)  | 792.26 (0.06) a, A                | 767.57 (0.96) a, B  | 862.13 (0.00) a, A                  | 826.47 (0.01) a, B | 1372.53 (2.56) a, A               | 1366.03 (0.78) a, B |
| CW - 16 % - 24 h | 16.10 (0.12)              | 16.73 (0.03)  | 787.65 (0.73) b, A                | 762.55 (1.09) bc, B | 847.61 (0.04) b, A                  | 819.77 (0.08) b, B | 1366.07 (0.47) b, A               | 1356.47 (1.79) b, B |
| CW - 18 % - 24 h | 18.09 (0.08)              | 17.92 (0.53)  | 782.15 (1.23) c, A                | 741.30 (0.21) f, B  | 832.55 (0.04) c, A                  | 793.88 (0.12) e, B | 1351.17 (1.21) e, A               | 1345.07 (0.74) c, B |
| HW - 16 % - 12 h | 15.73 (0.07)              | 16.24 (0.11)  | 786.41 (0.67) b, A                | 765.20 (0.76) ba, B | 847.55 (0.06) b, A                  | 819.93 (0.13) b, B | 1364.80 (2.18) cb, A              | 1354.87 (3.35) b, B |
| HW - 16 % - 18 h | 15.33 (0.08)              | 15.75 (0.20)  | 788.12 (1.19) b, A                | 764.83 (1.46) ba, B | 847.58 (0.06) b, A                  | 819.87 (0.03) b, B | 1364.40 (2.27) cb, A              | 1354.07 (2.68) b, B |
| HW - 16 % - 24 h | 15.54 (0.06)              | 16.34 (0.08)  | 783.10 (0.63) c, A                | 761.65 (0.62) c, B  | 847.56 (0.05) b, A                  | 813.38 (0.06) c, B | 1360.93 (1.47) c, A               | 1353.77 (2.63) b, B |
| HW - 18 % - 12 h | 17.74 (0.53)              | 18.28 (0.04)  | 771.90 (1.07) d, A                | 758.35 (0.83) d, B  | 833.46 (0.09) c, A                  | 806.69 (0.00) d, B | 1355.87 (2.32) d, A               | 1342.77 (1.22) c, B |
| HW - 18 % - 18 h | 17.79 (0.56)              | 18.08 (0.11)  | 768.00 (0.34) f, A                | 754.51 (1.43) e, B  | 826.61 (0.01) d, A                  | 806.53 (0.11) d, B | 1354.90 (2.09) ed, A              | 1343.60 (2.77) c, B |
| HW - 18 % - 24 h | 17.78 (0.02)              | 18.37 (0.21)  | 769.01 (0.89) ef, A               | 752.45 (0.60) e, B  | 833.44 (0.08) c, A                  | 806.77 (0.25) d, B | 1354.53 (0.74) ed, A              | 1343.80 (1.90) c, B |
| S - 5s           | 16.89 (0.09)              | 15.98 (0.08)  | 770.64 (0.32) ed, A               | 761.36 (1.13) c, B  | 833.53 (0.04) c, A                  | 806.67 (0.01) d, B | 1351.07 (2.19) e, A               | 1345.63 (1.03) c, B |
| S - 10 s         | 21.19 (0.08)              | 21.17 (0.11)  | 763.30 (0.99) g, A                | 734.36 (0.40) g, B  | 793.81 (0.01) e, A                  | 781.36 (0.05) f, B | 1337.47 (1.42) f, A               | 1320.73 (1.45) d, B |
| S - 15 s         | 22.26 (0.13)              | 22.70 (0.07)  | 739.08 (0.81) h, A                | 732.53 (1.83) g, B  | 781.40 (0.06) f, A                  | 769.39 (0.20) g, B | 1325.13 (1.37) g, A               | 1313.37 (1.99) e, B |

<sup>[a]</sup> CW = cold water, HW = hot water, S = steam. Values in the parentheses are standard deviations. The same lower-case letter in the same column for the specific type of sorghum indicate no significant difference ( $p \leq 0.05$ ). The same upper-case letter in the same row for a given treatment and density type indicate no significant difference.

Table 3- 2 Porosity values of white and waxy white sorghum for different tempering treatments

| Treatment        | Porosity (%)  |               |
|------------------|---------------|---------------|
|                  | White         | Waxy White    |
|                  | Sorghum Grain | Sorghum Grain |
| Untempered Grain | 41.28 (0.11)  | 42.25 (0.09)  |
| CW - 16 % - 24 h | 42.14 (0.12)  | 43.78 (0.03)  |
| CW - 18 % - 24 h | 42.34 (0.07)  | 43.89 (0.04)  |
| HW - 16 % - 12 h | 42.38 (0.13)  | 43.52 (0.18)  |
| HW - 16 % - 18 h | 42.24 (0.17)  | 43.52 (0.11)  |
| HW - 16 % - 24 h | 42.46 (0.11)  | 43.74 (0.11)  |
| HW - 18 % - 12 h | 43.07 (0.16)  | 43.52 (0.11)  |
| HW - 18 % - 18 h | 43.35 (0.08)  | 43.84 (0.06)  |
| HW - 18 % - 24 h | 43.24 (0.09)  | 43.96 (0.08)  |
| S - 5s           | 43.96 (0.07)  | 43.42 (0.13)  |
| S - 10 s         | 44.29 (0.13)  | 44.29 (0.09)  |
| S - 15 s         | 44.74 (0.02)  | 44.54 (0.10)  |

### ***3.4.5. Abrasive Hardness Index***

The dehulling characteristics of the grains can be predicted using the abrasive hardness index, AHI (Zhao and Ambrose, 2017). AHI of the white and waxy white sorghum kernels varied from 21.4 - 23.7 and 21.4 - 24.6, respectively (Table 3-2). There was no significant difference in the AHI values between untempered and tempered kernels of white and waxy white sorghum. It implies that tempering method (cold water, hot water, and steam), moisture content, and tempering time had a negligible influence on the force required to abrade the outer surface of sorghum kernels. Additionally, there was no significant difference between the indices of white and waxy white kernels too, for both untempered and tempered kernels. The study by Ehiwe and Reichert (1987) on evaluating the dehulling quality of cowpea, pigeon pea, and mung bean cultivars using TADD reported that the seed's resistance to split (or its hardness index) and binding strength of the seed coat or pericarp to the seed cotyledon adversely affects the dehulling capacity of the seed. The results show that the adherence between the pericarp and endosperm is relatively stronger even after tempering. In other words, the tempering conditions in the study could not possibly impart a difference to the hardness between endosperm and pericarp. As explained by Zhao and Ambrose (2017), a higher AHI ensures that the bran can be brittle and clean separation of bran from the endosperm can be achieved. This might possibly influence the dehulling characteristics. Several other factors also affect dehulling capacity of sorghum like low disk abrasiveness of the grinding wheel in TADD which can be obtained by using grinding wheels with a smooth finish or increasing the grit size of the wheel (Mwasaru et al., 1988).

### ***3.4.6. Sorghum Kernel Characteristics***

#### ***3.4.6.1. Hardness Index***

The results of hardness index are tabulated in Table 3-2. It is evident that the mean kernel hardness of the waxy white sorghum kernels is higher when compared to the white sorghum kernels. The tempering conditions (cold water, hot water, and steam) also relatively influences the hardness index values. Hardness index of waxy white sorghum decreased from 80.93 to 79.24N when the tempering moisture content of coldwater treatment increased from 16 to 18% (w.b). In case of white sorghum, there was no significant difference in the HI values upon cold water tempering. Similar trend was observed on the harness index values of hot water tempered kernels too. With increase in tempering moisture content, the hardness decreased. The influence of tempering time on hardness was evident for hot water tempering of white sorghum, but not for waxy white kernels. This could be related to the composition of the sorghum kernels. The waxy white sorghum kernels have more starch content when compared to white sorghum kernels. Steam tempering also decreased the hardness index of sorghum kernels. With increase in tempering time the moisture content of the kernels increased and thereby decreased the hardness index. For instance tempering the white kernels at 20 psi for 5, 10 and 15 s resulted in the final moisture content of 16.9, 21.2 and 22.3% (w.b), respectively. The corresponding hardness indices are 73.7, 70.8, and 56.3 N. Similar trends, i.e., the negative correlation between the moisture content and grain hardness has been reported for wheat kernels (Tester, 1980), pomegranate seeds (Kingsly et al., 2006), and sorghum (Mwithiga and Sifuna, 2006; Zhao and Ambrose, 2017). The grains resistance to crushing was negatively influenced by moisture content. As mentioned earlier, the steam tempering had significantly reduced the hardness of kernels. Similar results for the red

sorghum explained that heat treatment mellows the grain kernels and thereby improve the break flour yield.

Untempered waxy white sorghum had greater hardness index than untempered white sorghum. This has caused all tempered waxy white sorghum from every treatment to have greater hardness index than the contemporary white sorghum. In a comparative study by Patwa et al. (2014) between hard and soft wheat classes, the hardness index of soft wheat varieties was lower than the hard wheat varieties by nearly three times. This difference was associated with the complex and tightly packed cell structure and, starch-protein matrix of hard wheat varieties. This could possibly be the reason for the discrepancy between the hardness index of waxy white and white sorghum. The greater hardness index of waxy white sorghum than white sorghum could be due to its tougher cell structure than white sorghum kernel.

#### ***3.4.6.2. Mean Kernel Diameter***

The mean kernel diameter of steam tempered white sorghum was greater than the untempered kernels, while all tempered waxy white sorghum kernels showed greater mean kernel diameter than the untempered kernels. The rise in mean kernel diameter is due to water penetration into the intracellular spaces of the kernel during tempering (Solomon and Zewdu, 2009). White and waxy white sorghum did not show any significant trend in its mean kernel diameter with increasing moisture content when treated with cold water. However, it increased with increase in moisture content when tempered with hot water. The mean kernel diameter increased from 2.95 to 3.45 mm (for white) and 3.59 to 4.16 mm (for waxy white) when tempered with steam at 20 psi for 15s. During the process the moisture content of white and waxy white sorghum increased to 22.3% (w.b.) and 22.70 % (w.b.) respectively. This proves that moisture content has a significant

effect on kernel diameter. The radial dimensions of mung bean was also reported to increase with increase in moisture content 9.9 to 18.3% (w.b.) (Ravikanth et al., 2013). Similar results were reported for sorghum (Mwithiga and Sifuna, 2006), white and black beans (Senthilkumar et al., 2018), coriander seeds (Coşkuner and Karababa, 2007). Untempered waxy white sorghum showed significantly greater mean kernel diameter than untempered white sorghum. This initial factor had caused waxy white sorghum to have greater mean kernel diameter when compared to white sorghum on addition of water during tempering in every treatment.

#### ***3.4.6.3. Mean Kernel Weight***

The mean kernel weight of untempered white and waxy white sorghum was 24.29 and 36.07 mg, respectively. Tempering of the kernels increased the mean kernel weight of the kernels. This is due to the penetration of water to the intercellular spaces of the kernels. From the data in table 3-2 it is evident that steam tempered waxy white sorghum kernels have relatively greater mean kernel weight when compared to all the other tempering conditions. Temperature of tempering water and time had not significantly influenced the weight of white and waxy white sorghum kernels. Steam tempering had increased the final moisture of the kernels. Steam tempering at 20psi for 15s increased the moisture content of white sorghum from 13.5 to 22.3% (w.b) whereas for waxy white kernels the corresponding change was from 13.3 to 22.7% (w.b). This increased the kernel weight by 7.45% for white sorghum while the corresponding increase in waxy white kernels was 10.86% (Table 3-2). As the mean kernel weight of the kernels does not show any significant differences with respect to moisture content, it throws light upon the influence of steam tempering on kernel characteristics. More water penetration into the kernels is achieved by steam tempering than other methods. The mean kernel weight of millet increased on tempering from 5% to 25% (dry basis (d.b)) (Baryeh, 2002), whereas for hemp seeds it was reported to

decrease from 15.3 g at 8.62% d.b. to 16.9 g at 20.84% d.b. (Sacilik et al., 2003). The initial high mean kernel weight of untempered waxy white sorghum than untempered white sorghum had led the mean kernel weight of the former to be greater than the latter after conditioning in all the treatments. This could be related to the composition of the sorghum kernels. The waxy white sorghum kernels have more starch composition when compared to white sorghum kernels.

Table 3- 3. Single Kernel Characterization Properties and Abrasive Hardness Index of white and waxy white sorghum for different tempering treatments <sup>[b]</sup>

| Treatment        | Abrasive hardness Index |                   | Mean Kernel Hardness Index |                    | Mean Kernel Diameter (mm) |                  | Mean Kernel Weight (mg) |                     |
|------------------|-------------------------|-------------------|----------------------------|--------------------|---------------------------|------------------|-------------------------|---------------------|
|                  | White                   | Waxy White        | White                      | Waxy White         | White                     | Waxy White       | White                   | Waxy White          |
|                  | Sorghum Grain           | Sorghum Grain     | Sorghum Grain              | Sorghum Grain      | Sorghum Grain             | Sorghum Grain    | Sorghum Grain           | Sorghum Grain       |
| Untempered grain | 22.65 (1.72) a, A       | 21.43 (0.00) a, A | 73.18 (0.78) a, B          | 81.52 (0.67) a, A  | 2.95 (0.01) d, B          | 3.59 (0.01) d, A | 24.29 (0.27) b, B       | 36.07 (1.12) b, A   |
| CW - 16 % - 24 h | 22.25 (1.17) a, A       | 22.25 (1.17) a, A | 71.23 (1.47) abcd, B       | 80.93 (1.39) ab, A | 3.07 (0.01) cd, B         | 3.86 (0.05) c, A | 25.44 (0.11) bac, B     | 36.40 (0.00) bc, A  |
| CW - 18 % - 24 h | 21.43 (0.00) a, A       | 21.32 (0.15) a, A | 71.41 (0.24) abc, B        | 79.24 (0.11) ab, A | 3.11 (0.02) c, B          | 3.82 (0.02) c, A | 24.86 (0.41) bac, B     | 36.54 (1.08) bc, A  |
| HW - 16 % - 12 h | 23.60 (0.00) a, A       | 22.25 (1.17) a, A | 71.03 (0.11) abcd, B       | 79.91 (1.19) ab, A | 3.06 (0.05) cd, B         | 3.81 (0.02) c, A | 24.34 (0.59) bac, B     | 37.43 (0.28) bac, A |
| HW - 16 % - 18 h | 23.60 (0.00) a, A       | 24.60 (1.42) a, A | 71.75 (0.57) ab, B         | 78.99 (1.43) ab, A | 3.06 (0.02) cd, B         | 3.82 (0.03) c, A | 24.47 (0.55) bac, B     | 36.43 (0.21) bc, A  |
| HW - 16 % - 24 h | 23.34 (0.37) a, A       | 24.60 (1.42) a, A | 71.97 (0.00) ab, B         | 79.43 (0.08) ab, A | 3.06 (0.05) cd, B         | 3.84 (0.02) c, A | 24.56 (0.75) bac, B     | 36.44 (0.43) bc, A  |
| HW - 18 % - 12 h | 22.13 (0.99) a, A       | 23.60 (0.00) a, A | 69.47 (1.30) bcd, B        | 79.01 (1.44) ab, A | 3.25 (0.04) b, B          | 3.95 (0.03) b, A | 25.89 (0.16) ba, B      | 37.99 (1.43) bac, A |
| HW - 18 % - 18 h | 21.43 (0.00) a, A       | 22.51 (1.53) a, A | 68.82 (0.56) bcd, B        | 78.11 (0.93) b, A  | 3.24 (0.05) b, B          | 3.95 (0.02) b, A | 25.13 (0.02) bac, B     | 37.32 (0.72) bac, A |
| HW - 18 % - 24 h | 23.73 (0.19) a, A       | 23.73 (0.19) a, A | 68.27 (0.00) cd, B         | 77.99 (0.33) b, A  | 3.23 (0.00) b, B          | 3.98 (0.01) b, A | 25.23 (0.19) bac, B     | 37.1 (1.18) bac, A  |
| S - 5s           | 21.43 (0.00) a, A       | 22.51 (1.53) a, A | 68.01 (1.32) d, B          | 78.06 (1.18) b, A  | 3.13 (0.01) cb, B         | 3.80 (0.04) c, A | 24.77 (0.29) bac, B     | 37.68 (1.01) bac, A |
| S - 10 s         | 21.32 (0.16) a, A       | 22.54 (1.50) a, A | 62.03 (1.47) e, B          | 71.72 (0.91) c, A  | 3.09 (0.04) c, B          | 3.97 (0.01) b, A | 23.85 (0.70) ba, B      | 38.83 (0.36) c, A   |
| S - 15 s         | 22.37 (1.01) a, A       | 23.73 (0.18) a, A | 56.43 (0.48) f, B          | 65.01 (0.26) d, A  | 3.45 (0.06) a, B          | 4.16 (0.01) a, A | 26.10 (0.61) a, B       | 39.99 (0.52) a, A   |

<sup>[b]</sup> CW = cold water, HW = hot water, S = steam. Values in the parentheses are standard deviations. The same lower-case letter in the same column for the specific type of sorghum indicate no significant difference ( $p \leq 0.05$ ). The same upper-case letter in the same row for a given treatment and property type indicate no significant difference ( $p \leq 0.05$ ).



### ***3.4.7. Coefficients of Friction***

#### ***3.4.7.1. Static Coefficient of Friction***

Untempered kernels of white sorghum had a static coefficient value of 0.17 whereas the same for waxy white was 0.24. The coefficient of friction values for white sorghum kernels varied from 0.17 - 0.29 and waxy white from 0.24 - 0.35 for static friction (Table 3-3). The highest value for coefficient of static friction, 0.35, was exhibited by steam tempered waxy white kernels. Although, the static coefficient of friction increased with increase in moisture content for both the kernels there was no specific trend observed in the values. Tempering had a more pronounced effect on the static friction coefficient values in case of white sorghum kernels when compared to the waxy white sorghum kernels. Duration of tempering or heat treatment did not significantly influenced the coefficients in both white and waxy sorghum. Steam treatment at 20 psi from 5s to 15s raised the static coefficient of friction from 0.25 to 0.35 in waxy white sorghum. Increasing the treatment time had positively increased the moisture content from 16.89 % (w.b.) to 22.26 % (w.b) and thus the static coefficient of friction. Greater amount of water present in the kernels tempered with steam for 15s possibly increased the adhesive forces between the kernel and the surface of galvanized steel, causing it to have a high coefficient of static friction (Balasubramanian and Viswanathan, 2010; Shirkole et al., 2011). Rise in static coefficient of friction when millet was tempered from 5 to 22.5% (db) was reported by Baryeh (2002). Similar trend in this frictional property against moisture content was also reported in pearl millet (Jain and Bal, 1997), fenugreek (Altuntaş et al., 2005) and hemp seed (Sacilik et al., 2003). Kernels tempered with steam at 20 psi for 15s also had the highest static coefficient of friction when compared to kernels from all treatments. This is also attributed to the highest moisture content of kernels of that respective

treatment. However, attributing the increasing static coefficient of friction of the kernels with increasing moisture content when the steam tempering time increased from 5s to 15s may not be the only cause for the increment in the static coefficient of friction. Surface characteristics of the kernel could have also possibly played a role in the increment. Hence, it is important to know the surface structure of both white and waxy white sorghum kernels.

#### ***3.4.7.2. Rolling Coefficient of Friction***

Steam tempered and untempered white sorghum displayed the highest (0.32) and lowest (0.24) rolling coefficient of friction respectively when compared to other treatments (Table 3-3). Steam tempered (0.29) waxy white sorghum exhibited higher rolling coefficient of friction when compared to untempered (0.24) and hot water tempered kernels (0.243). The rolling coefficient of friction increased with increment in the moisture content when white and waxy white sorghum were treated with cold and hot water. The results agreed with the results obtained from coefficient of rolling friction studies on coriander seeds (Coşkuner and Karababa, 2007) fenugreek (Altuntaş et al., 2005), rape seed (Çalışır et al., 2005) and millet (Baryeh, 2002) against galvanized sheet. Coşkuner and Karababa (2007) explained the increment in rolling coefficient of friction in coriander seeds with increasing moisture. According to the authors, the increase in cohesive forces developed between the kernel and surface on addition of water during tempering made the seeds rougher than usual and reduced its sliding characteristics. No significant trend was observed in the rolling coefficient of friction with increasing heat treatment time in white and waxy white sorghum. However, a distinct increase was observed in this frictional property when white and waxy white sorghum were steam tempered at 20 psi from 5 to 15 s. The hollow cylinder containing the grains was removed for the sample to form a cone on the flat galvanized steel plate before the plate was inclined to calculate the rolling coefficient of friction. From, the angle of repose results

(Table 3-3), steam tempered (for 15s) kernels displayed greatest angle of repose, which can be attributed to the reduced flowability between the kernels itself causing greater force required to overcome the flowability between the kernels and overcome the cohesive forces between the kernels and the surface. White sorghum tempered with steam at 20 psi for 15s (0.32) exhibited the highest rolling coefficient of friction when compared to all other treatments on white sorghum. While, waxy white sorghum tempered with the same steam condition for 15s (0.29) and 10s (0.28) and, with cold water for 24 hours, 18% (w.b.), (0.29) showed the highest rolling coefficient of friction when compared to all other treatments.

White sorghum had either greater or insignificantly different rolling coefficient of friction when compared to waxy white sorghum for a given treatment. Since, friction is a force existing between two surfaces in contact, this difference could possibly be ascribed to the seed coat structure of white sorghum kernels. Tempering could have possibly made the outer surface of white sorghum kernels rougher than for waxy white sorghum, due to which the former showed greater resistance to sliding (high rolling coefficient of friction) than the latter (low rolling coefficient of friction).

#### ***3.4.8. Filling Angle of Repose***

Steam tempered and untempered white and waxy white sorghum showed the highest and lowest angle of repose respectively when compared to each other (Table 3-3). Thus, a sharp difference in moisture content between these two treatments explained that moisture content of kernel significantly affects angle of repose. Cold water tempered white sorghum displayed a decreasing trend in angle of repose with increasing moisture content, while an opposite trend was observed in waxy white sorghum. However, both white and waxy white sorghum showed a

significant increase in this property with increment in moisture content when tempered with hot water. Similar trend of increment in angle of repose with moisture content was reported in *Sericea Lespedeza* seeds (Mahapatra et al., 2019), alfalfa seeds (Togo et al., 2018) and teff seeds (Zewdu and Solomon, 2007). The angle of repose of coriander seeds was reported to increase significantly from 24.9° to 30.7° when the moisture content increased from 7.10 to 18.94 % d.b. No significant trend was observed in both with time of heat and steam treatment. According to Irtwange and Igbeka (2002) and Shirkole et al., 2011, tempering of grains increases the plasticity effect on the seed surface which causes the seeds to stick to each other. This improves the stability, reduces the flowability and increases the angle of repose.

Untempered white sorghum kernels, white sorghum kernels treated with cold water (final m.c. 18% w.b), hot water (final m.c. 18% w.b) for 18 hours and steam at 20 psi for 15s had significantly lower angle of repose than the corresponding treatments on waxy white sorghum kernels (Table 3-3). However, white sorghum kernels treated with hot water (final m.c. 16% w.b) for 24 hours had significantly higher angle of repose than the corresponding treatment in waxy white sorghum kernels. All other treatments have no significant difference between white and waxy white sorghum kernels.

Table 3- 4. Angle of repose and Coefficients of Friction of white and waxy white sorghum for different tempering treatments <sup>[c]</sup>

| Treatment        | Angle of Repose (°) |                   | Coefficient of Static Friction |                   | Coefficient of Rolling Friction |                   |
|------------------|---------------------|-------------------|--------------------------------|-------------------|---------------------------------|-------------------|
|                  | White               | Waxy White        | White                          | Waxy White        | White                           | Waxy White        |
|                  | Sorghum Grain       | Sorghum Grain     | Sorghum Grain                  | Sorghum Grain     | Sorghum Grain                   | Sorghum Grain     |
| Untempered grain | 17.54 (0.36) d, B   | 19.46 (0.60) b, A | 0.17 (0.06) c, B               | 0.24 (0.06) b, A  | 0.24 (0.01) d, A                | 0.23 ± 0.02 d, A  |
| CW - 16 % - 24 h | 19.45 (0.23) dc, A  | 19.54 (0.09) b, A | 0.23 (0.04) cb, A              | 0.25 (0.03) ba, A | 0.25 (0.01) d, A                | 0.23 ± 0.01 d, B  |
| CW - 18 % - 24 h | 18.77 (0.34) d, B   | 19.95 (0.33) b, A | 0.23 (0.06) cb, B              | 0.33 (0.05) ba, A | 0.27 (0.00) c, B                | 0.25 ± 0.00 bc, A |
| HW - 16 % - 12 h | 19.03 (0.31) d, A   | 20.10 ± 0.54 b, A | 0.27 (0.00) b, A               | 0.28 (0.06) ba, A | 0.25 (0.01) d, A                | 0.24 (0.00) cd, B |
| HW - 16 % - 18 h | 19.44 (0.83) dc, A  | 20.05 ± 0.54 b, A | 0.25 (0.03) b, A               | 0.24 (0.04) b, A  | 0.26 (0.01) d, A                | 0.23 (0.02) d, B  |
| HW - 16 % - 24 h | 21.02 (1.03) bc, A  | 19.54 ± 0.44 b, B | 0.28 (0.03) b, A               | 0.25 (0.06) ba, A | 0.25 (0.01) d, A                | 0.24 ± 0.01 cd, B |
| HW - 18 % - 12 h | 21.30 (1.01) bac, A | 22.20 (0.40) a, A | 0.28 (0.03) b, A               | 0.29 (0.06) ba, A | 0.27 (0.01) cb, A               | 0.25 (0.01) bc, B |
| HW - 18 % - 18 h | 21.50 (0.87) bac, B | 22.91 (0.89) a, A | 0.27 (0.00) b, B               | 0.35 (0.06) a, A  | 0.28 (0.01) cb, A               | 0.26 (0.01) b, A  |
| HW - 18 % - 24 h | 21.57 (0.94) ba, A  | 22.30 (0.34) a, A | 0.28 (0.00) b, A               | 0.32 (0.03) ba, A | 0.29 (0.01) b, A                | 0.26 (0.00) b, B  |
| S - 5s           | 21.49 (1.68) bac, A | 23.11 (0.83) a, A | 0.29 (0.04) b, A               | 0.25 (0.00) ba, A | 0.29 (0.01) b, A                | 0.25 (0.01) bc, B |
| S - 10 s         | 21.59 (1.20) ba, B  | 23.34 (0.56) a, A | 0.29 (0.04) b, A               | 0.31 (0.04) ba, A | 0.29 (0.01) b, A                | 0.28 (0.01) a, A  |
| S - 15 s         | 23.20 (0.95) a, A   | 23.60 (1.50) a, A | 0.29 (0.04) b, A               | 0.35 (0.00) a, B  | 0.32 (0.01) a, A                | 0.29 (0.01) a, B  |

<sup>[c]</sup>CW = cold water, HW = hot water, S = steam. Values in the parentheses are standard deviations. The same lower-case letter in the same column for the specific type of sorghum indicate no significant difference ( $p \leq 0.05$ ). The same upper-case letter in the same row for a given treatment and property type indicate no significant difference ( $p \leq 0.05$ ).

### 3.5. Conclusions

1. The bulk and tapped density of both white and waxy white decreased with increasing moisture content when tempered with cold water, hot water, and/or steam. Sorghum kernels tempered with steam at 20 psi for 15s had the lowest bulk and tapped density. White sorghum had greater bulk and tapped density than waxy white sorghum under all conditions.
2. Moisture content or tempering condition did not have any significant effect on the abrasive hardness index of both white and waxy white sorghum. No significant difference was either observed in the abrasive hardness index values between white and waxy white sorghum for any treatment.
3. The hardness index of both white and waxy white sorghum decreased with increasing moisture content and tempering time when treated with steam. Waxy white sorghum kernels at all the tempering conditions had higher hardness index values than white sorghum kernels.
4. Mean kernel diameter increased significantly when kernels were tempered with steam. Waxy white sorghum had a greater mean kernel diameter than white sorghum.
5. The mean kernel weight of both white and waxy white increased when kernels were tempered with steam. Waxy white sorghum had a greater mean kernel weight than all untempered and tempered white sorghum kernels.

6. Static coefficient of friction of both white and waxy white sorghum increased when tempered with steam. A distinct trend was not observed between the static coefficient of friction of white and waxy white sorghum kernels.
7. The rolling coefficient of friction of both white and waxy white sorghum increased with steam tempering. Cold water tempered waxy white sorghum kernels also displayed high rolling coefficient of friction when compared to hot water and untempered kernels. White sorghum exhibited significantly similar coefficient of rolling friction as that of waxy white sorghum kernels.
8. The angle of repose of both white and waxy sorghum increased with steam and hot water tempering. An evident trend between the angle of repose of white and waxy white sorghum kernels was not displayed with tempering.

The significant effect of conditioning used (cold water, hot water or steam) on all physical properties was more evidently observed between untempered and steam tempered kernels. Though steam tempering of sorghum at 20 psi for 15s showed the desired physical properties in kernels like lower bulk density, higher angle of repose and coefficients of friction and, lower hardness index for efficient handling, transportation and processing; the high moisture content of sorghum from this treatment is unsafe and risky for long-term storage. Though steam tempered kernels require the least time to temper, proper drying techniques and storage conditions are necessary to maintain the quality of kernels. Prospective studies can be conducted on tempering white and waxy white sorghum to 19% m.c. (w.b) using cold water and hot water to obtain better results in physical properties.

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## **Chapter 4 – EFFECTS OF TEMPERING ON WHITE AND WAXY WHITE SORGHUM MILLING AND FLOUR PROPERTIES**

### **4.1. Abstract**

*Sorghum is widely cultivated in the major countries of the world and having several advantages in terms of nutrition, easy cultivation and absence of gluten. However, inconsistent flour production, discoloration of flour due to bran contamination and high starch damage have been associated with widely followed milling techniques on sorghum. This research involved the development of an efficient roller milling technique using the laboratory-scale roller mill to produce white and waxy white sorghum flour. The developed roller milling flowsheet consisted of four break rolls and eight reduction rolls. 11 tempering conditions using cold water, hot water and steam were applied on both types of sorghum. Steam tempering the sorghum kernels to 15s removed maximum bran (10.12% and 7.17% respectively) and produced flour with lowest ash content (0.66% and 1.16% respectively), brightest color (L values: 87.48 and 82.09 respectively) and lowest crude fat content (0.20% and 1.96% respectively), although it reduced the protein content (7.61% and 8.58% respectively) and flour yield (57.91% and 31.80% respectively) heavily. However, white and waxy white sorghum tempered with cold water for 24 hours to bring to final moisture content 18% (wet basis) produced good flour yield (62.08% and 40.50% respectively) and bran removal (6.55% and 6.42% respectively) without compromising the protein content (8.55% and 9.72% respectively), ash content (0.63% and 0.94% respectively), crude fat content (0.42% and 0.72% respectively) and produced flour with the lowest starch damage (5.25%), proving this tempering method could be more suitable to roller mill white and waxy white sorghum. The developed milling flowsheet produced excessive shorts from waxy white sorghum*

*ineffective of the tempering method. 98% of total flour produced from all tempering conditions had particle size of less than 212  $\mu\text{m}$ .*

**Keywords:** roller mill, tempering, flour yield, bran, ash, protein, particle size

## 4.2. Introduction

Cereal grains like wheat, maize, rice and others have been our major source of food since man started farming. Sorghum ranks fifth in importance after maize, rice, wheat, and barley. Countries like the United States of America, Mexico, Brazil, Australia, Italy and France were reported to have the highest sorghum yields (approximately >4 tons/ha) when compared to other countries due to the practice of intensively mechanized sorghum farming (Taylor and Duodu, 2018). In most of Africa (except Southern Africa), Central America and Saudi Arabia, sorghum accounts for >10% of the total cereal production (Taylor and Duodu, 2018). FAOSTAT (2017) states sorghum as one of the top 20 commodities produced in USA and its Gross Production Value had increased from 1,000k dollars to 1,800k dollars from 2006 to 2016 (FAOSTAT, 2016) with most of it being used for ethanol production and animal feed (Wang et al., 2000). Sorghum being a hardy crop has low water requirements and is drought tolerant. These characteristics make it very suitable to be cultivated in regions that are adversely affected by climate change (Padulosi et al., 2009; Hadebe et al., 2017). Sorghum is also a C<sub>4</sub> tropical-type plant which makes it more capable than C<sub>3</sub> plants like wheat and its contemporaries in employing high solar energy in tropical latitudes (Sage and Zhu, 2009). Hence, sorghum can be widely cultivated in regions with high incident solar energy.

Celiac Disease (CD) is a permanent food intolerance caused by ingestion of gluten from wheat, rye and barley which harms the enterocytes and causes malabsorption of significant

nutrients by the intestine (Holmes et al., 2009). The existence of CD was underreported for a long time. Although, now it is known to be one of the most common food intolerances around the world (Catassi and Fasano, 2009). It reportedly affects 0.7% of the total U.S. and European populations and is also prevalent in North Africa, the Middle East and India (Catassi and Fasano, 2009). A gluten-free (GF) diet consisting of foods like vegetables, fruits, meat, and GF products is the only remedy to CD till date (Morreale et al., 2018). This demand has boomed the global GF market. The production of GF foods in the US has increased around by 136% from 2013 to 2015, reaching an approximate value of \$11 billion (Foschia et al., 2016). While in Europe, the GF market is expected to have a growth by approximately 10% until 2019 (Elli et al., 2015). Sorghum being GF could be a suitable substitute to gluten containing foods and growing gluten-free market.

Although sorghum has considerable benefits, the commercialization of the sorghum products is limited due to the lack of standard milling processes. The technology of milling sorghum is not as developed as that of wheat, rice or maize (Kebakile, 2008; Beta et al., 2000). Traditional methods of hand pounding and dehulling followed by hammer milling are still widely used for producing sorghum flour (Taylor, 2003). These methods result in coarser flour with low ash and oil content (Kebakile, 2007). Moreover, abrasive decortication-hammer milling (ADHM) result in higher endosperm loss during bran removal (Kebakile et al., 2008). Structural similarity of sorghum kernels with that of corn, encouraged wet milling of sorghum like corn (Adeyemi, 1983). However, its smaller kernel size when compared to corn did not support wet milling and resulted in low extraction rate and loss of starch (Watson, 1984). In addition, the fragile and friable pericarp of sorghum caused it to easily break during wet milling and produced specks of bran in the milled flour (Wang et al., 2000).

The similarity in kernel size of sorghum with that of wheat motivated the researchers, Anderson et al. (1969); Kebakile et al. (2008) to study the impact of roller milling on sorghum. Kebakile et al. (2008) utilized two pairs of roller mills to mill sorghum. The first pair of rolls broke the sorghum kernels to smaller fragments with the bran intact. The second pair of rolls ground the fragments to flour by separating it from bran particles. The research also compared the performance of roller milling sorghum with ADHM where the former resulted in higher production rate of fine-grained flour with greater oil, ash, and protein content than the latter. However, roller milled sorghum flour exhibited greater bran contamination when compared to sorghum flour produced from ADHM.

In order to overcome the limitation of bran contamination the effect of tempering on sorghum kernels were studied. Tempering, conditioning with water, is known to toughen the bran and soften the endosperm. Additionally, it facilitates proper separation of bran and endosperm during milling. Tempering of sorghum had positively influenced the flour extraction rate (Zinn et al., 2014) and particle size distribution of flour during ADHM (Adeyemi, 1983). The effect of different tempering conditions (cold water, hot water and steam tempering) on physical and mechanical properties of red sorghum kernels and its milling quality are reported by Zhao and Ambrose (2017; 2018).

As tempering affects physical properties and milling behavior of grain, it subsequently affects flour properties like particle size, composition, pasting properties and dough rheology (Zhao and Ambrose, 2017). Additionally, studies on particle size and density are necessary for various unit operations like separating, conveying, mixing and in designing of storage structures, pneumatic and other processing equipment (Plange et al., 2003; Raigar and Mishra, 2015). So, the objective of this study was to develop a milling flow sheet for white and waxy white sorghum and

also evaluate the effects of different tempering conditions on the physical and chemical properties of white and waxy white sorghum flour.

### 4.3. Materials and methods

#### 4.3.1. White and Waxy White Sorghum Sample Preparation

White and waxy white sorghum kernels were chosen for this study. 45.4 kg of each variety of sorghum free from foreign matter such as dust, dirt, stones and chaff were procured from Nu Life Market, Scott City, KS. The initial moisture content (m.c.) of white and waxy white sorghum kernels was measured using ASABE Standard S352.2 method (ASABE, 2012):

$$\text{moisture content (wet basis)} = \frac{m_w}{(m_w + m_d)} \times 100 \quad (1)$$

Where,  $m_w$  = the mass of water removed (g)

$m_d$  = the mass of sample after drying at 130 °C for 18h (g)

The initial moisture content (wet basis (w.b.)) of white and waxy white sorghum were found to be  $13.46 \pm 0.08$  % and  $13.29 \pm 0.16$  %, respectively

*Cold water tempering:* White and waxy white sorghum samples were tempered with predetermined amount of cold distilled water (water available at room temperature) (required water quantity was measured using eq 2 (Balasubramanian, 2001)). The tempering process was carried out in a rotating drum to bring them to the final moisture contents of 16 and 18% (w.b). These moisture contents used in this study were chosen based on the previous research conducted by Zhao et al. (2016) on red sorghum.

$$Q = \frac{W(M_f - M_i)}{(100 - M_i)} \quad (2)$$

Where,  $Q$  = amount of distilled water required for tempering (ml)

$W$  = the weight of sample required to be tempered (Kg)

$M_f$  = moisture content (% w.b) to which the grain must be tempered

$M_i$  = initial moisture content (% w.b) of sample

The cold water tempered samples were air tightly packed in zip lock bags. The zip lock bags containing the cold water tempered samples were held at room temperature for 24 h for uniform distribution of moisture throughout the samples.

*Hot water tempering:* The samples were treated with the pre-calculated amount of cold distilled water (based on eq 2) in the rotating tempering drums to bring them to final m.c. of 16 and 18% (w.b.). The treated samples were then filled into glass beakers and sealed with aluminum foil to prevent water evaporation. The beakers containing the treated samples were placed in hot water bath at 60 °C for 12, 18, and 24 h (Cecil, 1992). The glass beakers were shaken intermittently for every 30 min for moisture equilibration. After conditioning the samples for the respective time periods (12, 18, and 24 h), sample were cooled at room temperature prior to the evaluation of the kernel properties.

*Steam Tempering:* The samples were tempered with steam at 20 psi for 5, 10, and 15 s in a hollow container with a screw passing horizontally through the inside of the container. The screw was used to mix the sorghum samples as steam passed through the inlet which allows the uniform distribution of steam throughout the sample. After the samples were steam treated, the samples were spread on flat trays for about 8 h to evenly dry the samples, which was achieved by ambient air drying. The samples were then transferred to ziplock bags and stored in refrigerator at 5 °C until the experimental measurements.

### ***4.3.2. Sorghum Roller Milling***

Milling trials were conducted on laboratory-scale tabletop roller mill (Ross, Oklahoma City, OK.) in Department of grain Science and Industry, Kansas State University. Preliminary trials were performed based on the understanding of milling basics and following a trial and error approach using tempered white and waxy white sorghum kernels.

Briefly, milling process comprises of set of rollers and sifters. The two types of rollers for milling are break rolls and reduction rolls. The purpose of break rolls is to break open the kernels and scrap off the endosperm from the bran. The reduction rolls serve to reduce the endosperm into flour. The developed flow sheet (Fig 4-1) for roller milling of sorghum consisted of four pairs of break rolls and eight pairs of reduction rolls along with sieving. The first two pairs of break rolls broke the kernel to smaller particles keeping the bran intact, and the last two break rolls scrapped the bran from the endosperm. Based on the particle size, the grits were classified into fine and coarse. The classified grits were reduced to flour using respective smooth rolls. Among the reduction rolls, three pairs of reduction rolls were used for milling coarse grits and five were used for milling fine grits. The milling outcomes from each pair of rolls was passed through a set of sifters (Fig. 4-1.) and the milling stocks from each sieve were collected and weighed to separate the outcomes based on their size.

The break rolls had a speed differential of 2.5:1 and roll disposition of dull-to-dull whereas, speed differential of the reduction rolls was maintained at 1.25:1. The roll gaps for break and reduction rolls were determined based on preliminary trials (Fig. 4-1). As efficient milling process ensures quantity and quality of the extracted flour; flour extraction yield, particle size distribution of the flour and ash content were analyzed for all the preliminary trials. The ash content determines the purity of produced flour and it varies with bran contamination. Based on the preliminary trials,

the selection criteria for flow sheet was narrowed down to flour extraction yield and ash content. The milling process was performed in triplicates for every tempering treatment on white and waxy white sorghum. The fractions collected from the milling process were bran, fine bran, shorts, red dog and flour. The milling fractions were collected as shown in Fig. 1. at the end of the milling process from each replicate of a tempering treatment and combined to calculate the yield (%) of each fraction. The yield (%) of each milling fraction was calculated using equation (3)

$$Yield (\%) = \frac{\text{Weight of total milling fraction obtained from milling (g)}}{\text{Initial weight of grain taken (g)}} \times 100 \quad (3)$$

The sieve sizes used for separating the milling outcomes are explained in table 4-1.

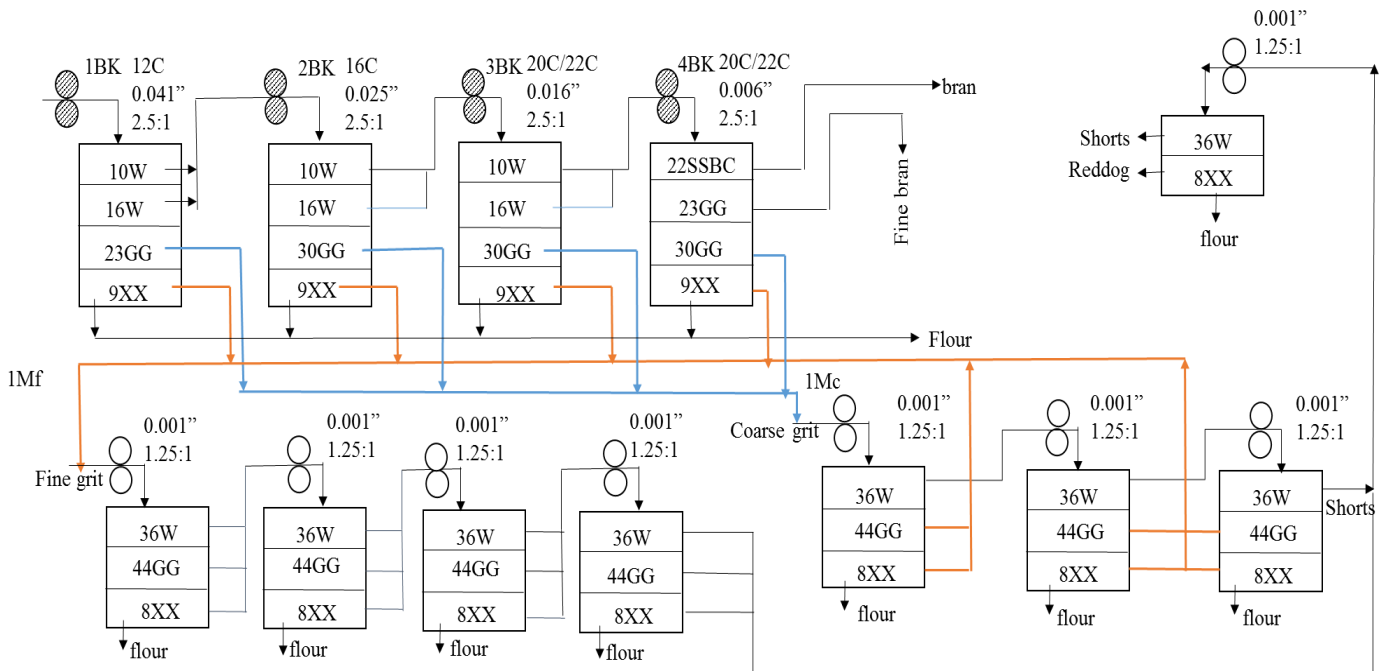


Fig 4- 1. Sorghum Roller Milling Flowsheet

(BK: Break, C: Corrugation, Mf: Fine grit milling, Mc: Coarse Grit Milling)



Table 4- 1. Sieve Sizes used in Fig. 4-1. for separating milling outcomes (W: wire, GG: grits gauze, SSBC: stainless steel bolting cloth, XX: flour silk)

| Sieve Used | Sieve opening ( $\mu\text{m}$ ) |
|------------|---------------------------------|
| 10W        | 2030                            |
| 16W        | 1180                            |
| 22SSBC     | 977                             |
| 23GG       | 900                             |
| 30GG       | 600                             |
| 36W        | 478                             |
| 44GG       | 425                             |
| 8XX        | 193                             |
| 9XX        | 150                             |

#### ***4.3.3. Flour Particle Size Distribution***

The particle size distribution of white and waxy white sorghum flour samples was determined by Ro-Tap analysis according to ASABE Standard S319.4 (ASABE Standards, 2008). The standard US series sieves were stacked on the decreasing order of their aperture size in a sieve shaker (Ro-Tap model RX-29, W.S. Tyler, Mentor, Ohio). The sieves used in the study were 6, 8, 12, 16, 20, 30, 40, 50, 70, 100, 140, 200, 270, and pan. The apertures of the sieves in the stated order decreased successively from 4760  $\mu\text{m}$  to 53  $\mu\text{m}$  for US sieve number 6 to 270 respectively. The weight of all the empty sieves had been recorded. Flour ( $100 \pm 0.1$  g) was added to the topmost sieve and has been sieved for 10 min. The mass of sample retained on each sieve was recorded and the particle size distribution curve was plotted for both the samples.

#### ***4.3.4. Flour Chemical Composition***

The proximate composition of the white and waxy white sorghum samples were determined following the standard procedures. AACC 76.33 using SDmatic® (Chopin Technologies, France) was followed for estimating the damaged starch in flour. This amperometric procedure is based on the principle that iodine absorption by flour depends on the amount of damaged starch present in it (Medcalf and Gilles, 1965). Total starch (%) of flour samples was analyzed using Megazyme Total Starch Assay Procedure (Megazyme International Ireland Ltd., Bray, Ireland) (AACCI Method 76-13.01). The total starch in the flour was evaluated by gelatinizing in the presence of thermostable alpha-amylase followed by amyloglucosidase hydrolysis to glucose. Protein (%), crude fat (%), crude fiber (%) and ash (%) of white and waxy white sorghum flour were analyzed by an external lab using AACC Method 46-30.01, AACC Method 30-25.01, AACC Method 32-10.01 and AACC Method 08-01.01 respectively.

#### ***4.3.5. Moisture Content of flour***

The moisture content of both milled white and milled waxy white sorghum flour was measured in wet basis using standard method, AACC 44-15.02 (AACC, 2011). Briefly, 2-3 g of the flour has been dried in a hot air oven at 135 °C for 2h. The difference in weight to the initial has been used to calculate the moisture content.

#### ***4.3.6. Bulk Density of flour***

The bulk density of white and waxy white sorghum flour was measured using Winchester cup arrangement (Seedburo Equipment Co., Des Plaines, USA). The flour was allowed to fall freely into a cup of known volume, 1 pint (1pint = 550.61cm<sup>3</sup>) from a feed hopper at a height of

10 cm from the mouth of the cup. The flour was allowed to fall into the cup until excess starts to overflow. The excess flour was scrapped off from the top by passing a scrapper in a zigzag motion. The bulk density of flour was calculated as the ratio of mass of flour in the cup to the volume of the cup.

#### ***4.3.7. Tapped density of flour***

Tapped density of white and waxy white sorghum flour was measured using Autotap Density Analyzer (Quantachrome Instruments, Boynton Beach, Fla). (100 ± 1) g of flour from each treatment was filled in the measuring cylinder of the Analyzer and tapped for 750 times. The tapped density was calculated as the ratio of mass of sample taken to the final volume after tapping.

#### ***4.3.8. True Density of flour***

A gas pycnometer (AccuPync II 1340, Micromeritics, Norcross, Ga.) was used to measure the true density of white and waxy white sorghum flour. Sample holder of the pycnometer was filled with flour till little below its brim and enclosed tightly in a chamber after measuring the mass of flour filled in the sample holder. Helium gas was diffused into the sample holder to fill its entire volume. The true density of the flour was determined as the ratio of mass of flour taken to the volume of flour occupied by in the sample holder.

#### ***4.3.9. Color analysis of flour***

Color of white and waxy white sorghum flour was measured using HunterLab MiniScan EZ 45/0° Spectrophotometer (Hunter Associates Laboratory, Inc.). The reflected wavelength of light from the flour sample was measured by the sensor in the spectrophotometer to express the

color values in three scales: L - scale, where a lower number (0-50) indicates dark and a higher number (51-100) indicates light; a\* – scale represents the redness-greenness scale, where positive a\* number indicates red and a\* negative number indicated green; and b\* – scale runs from yellow to blue range, where positive b\* number indicates yellow and negative b\* number indicates blue.

#### ***4.3.10. Statistical Analysis***

The study was a completely randomized design with 11 treatments (two cold water treatments (final m.c. 16% and 18% w.b.), six hot water treatments (final m.c. 16% and 18% w.b. for tempering times of 12, 18 and 24h for each final m.c.) and three steam tempering treatments at 20 psi for 5, 10 and 15s) each for white and waxy white sorghum. All experiments were conducted in triplicates. The statistical significance between the treatments were analyzed using PROC GLM in SAS (ver. 9.3, SAS Institute, Inc., Cary, N.C.). Based on ANOVA, significant differences ( $\alpha = 0.05$ ) between treatments was compared.

## **4.4. Results and discussion**

### ***4.4.1. White and waxy white sorghum milling fraction yield***

The primary goal of milling is to maximize the recovery of endosperm from the grain with minimal contamination from bran. The presence of bran imparts undesirable discoloration to the flour. Milling outcome from the developed flow sheet is tabulated in Table 4-1 and Table 4-2. The following sections details the average yield (%) of milling fractions obtained from milling white and waxy white sorghum using the prepared flowsheet.

#### ***4.4.1.1. Flour***

Flour yield varied between 58-62% for white sorghum whereas that of waxy white sorghum was between 30-47% (Table 4-1). In case of white sorghum, there was no significant ( $p < 0.05$ ) difference flour yield with increasing tempering moisture content or type of tempering. With increase in moisture content, the flour yield of waxy white sorghum displayed a downward trend for both cold and hot water tempering. Tempering for 24h with hot water considerably reduced the flour yield of waxy white sorghum when compared to the other time periods. The yield was limited to 37% under this tempering condition. It has to be noted that the lowest value for yield was reported by steam tempering. Steam tempering for 15s reduced the flour yield to 30% for waxy white sorghum.

#### ***4.4.1.2. Bran***

Bran extraction from the grain during milling was evidently increased with increasing moisture content when tempered with cold water, hot water or steam (Table. 4-2). Steam tempering for 10 and 15s provided the most efficient separation of bran from white and waxy white sorghum

grain using the milling process. This consequently made the flour obtained from the treatments to have the least bran contamination.

#### **4.4.1.3. Fine Bran**

Though fine bran is an insignificant quantity of milling fraction, it can potentially contaminate flour. Steam tempered (15s) white and waxy sorghum produced maximum extraction of fine bran when compared to other treatments (Table. 4-2).

#### **4.4.1.4. Shorts**

Shorts are a by-product of milling coarse and fine grits which are in the form of flaked endosperm containing some bran, flour and germ (Trappey et al., 2015). A negative linear relationship was observed between the rate of production of shorts and tempering moisture content when tempered with cold water (Table. 4-2). Indirect conditioning in hot water tempering did not provide any effect in the production of shorts for white sorghum. Neither was any impact on shorts with increasing tempering moisture content or time. The lowest amount of shorts were procured from white sorghum tempered with cold water for 24h with final m.c. 18% (w.b).

A contrasting trend was observed with the amount of shorts produced in waxy white sorghum milling. Though tempering moisture content and time did not influence the production of shorts in waxy white sorghum milling, steam tempering for 10 and 15s produced the greatest amount of shorts among all treatments. Similar trend was shown by white sorghum also. The greater production of shorts from waxy white sorghum when compared to white sorghum from all tempered conditions displayed that most of the waxy white endosperm was lost in the form of shorts.

#### ***4.4.1.5. Red dog***

Red dog is also a by-product of milling grain which consists of bran, germ and flour. Both white and waxy white sorghum displayed a decrease in the production of red dog with an increase in tempering moisture content on using cold water (Table. 4-2). However, no specific trend was observed with the same upon increasing the tempering time and moisture content of hot water tempering. The production of red dog from both white and waxy white sorghum reduced gradually when steam tempering time increased from 5s to 15s. Steam tempering to 15s exhibited the lowest value of 10.05% for white sorghum. Waxy white sorghum from most tempered conditions produced greater amount of red dog when compared to white sorghum. This could possibly be due to the fact similar to the production of shorts that the applied tempering conditions on waxy white sorghum had critically caused maximum endosperm to be converted into these by-products.

Table 4- 2. Flour yield of white and waxy white sorghum from the developed flowsheet <sup>[a]</sup>

| Treatment        | Flour yield (%)     |                    |
|------------------|---------------------|--------------------|
|                  | White               | Waxy White         |
|                  | Sorghum             | Sorghum            |
| CW - 16 % - 24 h | 59.87 (0.43) abc, A | 43.33 (0.50) bc, B |
| CW - 18 % - 24 h | 62.08 (0.04) ab, A  | 40.50 (0.87) cd, B |
| HW - 16 % - 12 h | 61.01 (0.89) ab, A  | 45.42 (1.81) ab, B |
| HW - 16 % - 18 h | 62.96 (0.97) a, A   | 47.33 (0.27) a, B  |
| HW - 16 % - 24 h | 60.71 (0.08) abc, A | 41.97 (2.31) c, B  |
| HW - 18 % - 12 h | 61.25 (1.41) ab, A  | 41.21 (0.80) c, B  |
| HW - 18 % - 18 h | 56.44 (1.87) d, A   | 38.43 (0.31) de, B |
| HW - 18 % - 24 h | 59.99 (0.16) abc, A | 37.37 (0.18) e, B  |
| S - 5s           | 61.59 (1.37) ab, A  | 40.56 (1.02) cd, B |
| S - 10 s         | 59.50 (0.63) bc, A  | 29.54 (0.66) f, B  |
| S - 15 s         | 57.91 (0.54) cd, A  | 31.80 (0.80) f, B  |

<sup>[a]</sup> CW = cold water, HW = hot water, S = steam. Values in the parentheses are standard deviations. The same lower-case letter in the same column for the specific type of sorghum indicate no significant difference ( $p \leq 0.05$ ). The same upper-case letter in the same row for a given treatment and density type indicate no significant difference.



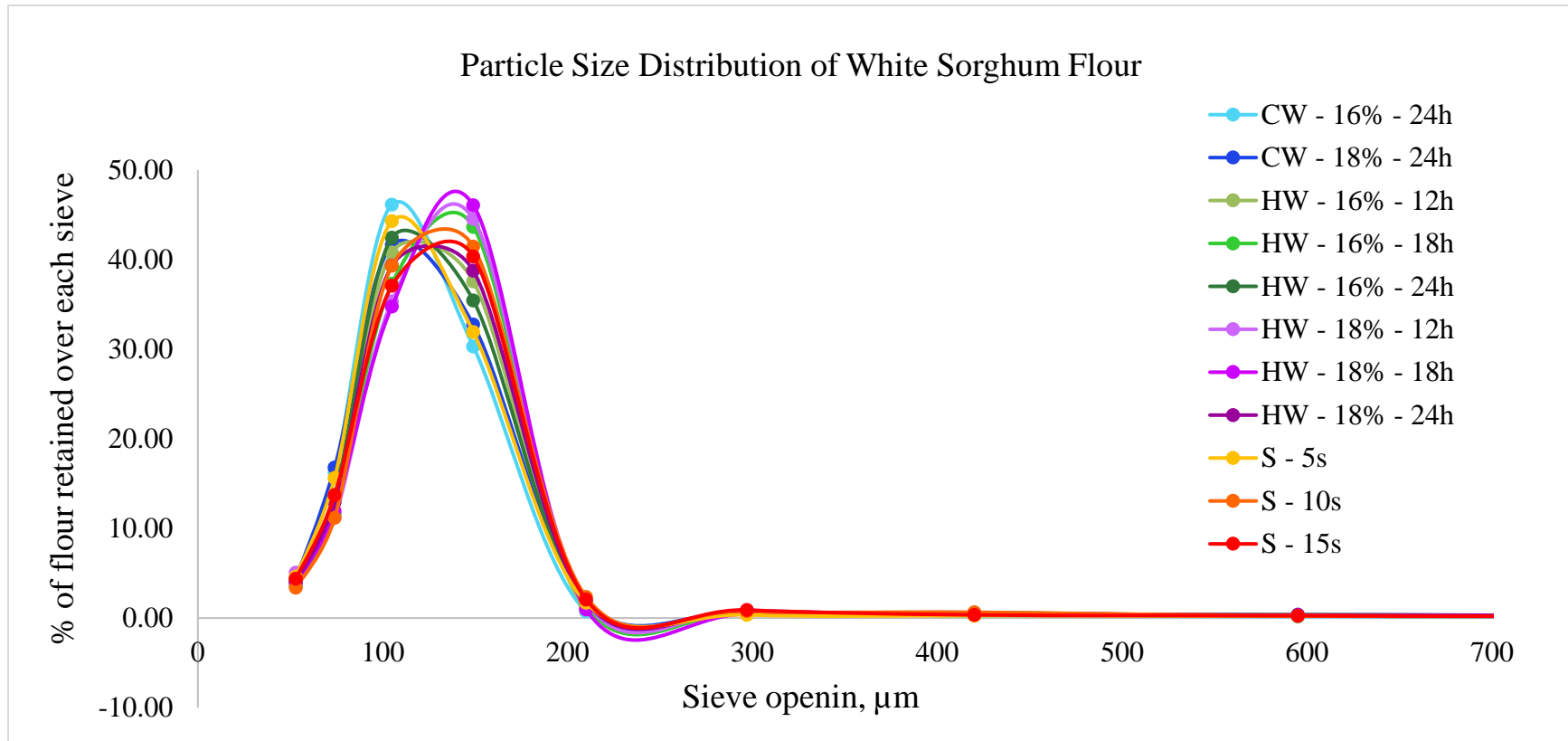
Table 4- 3. Milling outcomes of white and waxy white sorghum from the developed flowsheet <sup>[b]</sup>

| Treatment        | Bran (%)          |                    | Fine Bran (%)      |                    | Shorts (%)          |                    | Red dog (%)         |                    |
|------------------|-------------------|--------------------|--------------------|--------------------|---------------------|--------------------|---------------------|--------------------|
|                  | White Sorghum     | Waxy White Sorghum | White Sorghum      | Waxy White Sorghum | White Sorghum       | Waxy White Sorghum | White Sorghum       | Waxy White Sorghum |
| CW - 16 % - 24 h | 3.83 (0.18) fg, B | 5.40 (0.03) f, A   | 0.29 (0.05) f, B   | 0.53 (0.01) c, A   | 18.64 (0.69) ab, B  | 29.84 (0.68) d, A  | 12.59 (0.00) a, B   | 14.84 (0.02) bc, A |
| CW - 18 % - 24 h | 6.55 (0.21) c, A  | 6.42 (0.13) c, A   | 0.53 (0.01) de, A  | 0.56 (0.02) c, A   | 14.83 (0.13) e, B   | 32.07 (0.89) cd, A | 11.31 (0.55) bc, A  | 12.76 (0.26) f, A  |
| HW - 16 % - 12 h | 4.10 (0.17) f, B  | 4.84 (0.05) g, A   | 0.30 (0.07) f, A   | 0.29 (0.01) d, A   | 17.65 (0.12) abc, B | 30.11 (1.03) d, A  | 12.30 (0.40) ab, B  | 14.96 (0.58) bc, A |
| HW - 16 % - 18 h | 3.67 (0.13) g, B  | 5.57 (0.14) ef, A  | 0.28 (0.03) f, B   | 0.66 (0.01) b, A   | 17.46 (0.39) bc, B  | 26.93 (0.96) e, A  | 12.59 (0.55) a, B   | 14.50 (0.15) bc, A |
| HW - 16 % - 24 h | 3.58 (0.14) g, A  | 4.81 (0.09) e, B   | 0.59 (0.02) cde, A | 0.56 (0.02) c, A   | 18.86 (0.85) a, B   | 32.16 (0.78) cd, A | 11.88 (0.32) ab, B  | 14.92 (0.11) bc, A |
| HW - 18 % - 12 h | 5.43 (0.07) d, A  | 4.50 (0.06) h, B   | 0.70 (0.01) bc, A  | 0.69 (0.01) ab, A  | 16.67 (0.04) cd, B  | 32.20 (0.62) cd, A | 11.91 (0.01) ab, B  | 14.64 (0.06) bc, A |
| HW - 18 % - 18 h | 6.73 (0.02) c, A  | 5.53 (0.07) ef, B  | 0.62 (0.01) bcd, A | 0.69 (0.04) ab, A  | 16.94 (0.13) c, B   | 33.75 (0.49) bc, A | 11.54 (0.29) abc, B | 14.22 (0.18) cd, A |
| HW - 18 % - 24 h | 5.68 (0.14) d, B  | 6.09 (0.11) d, A   | 0.74 (0.02) b, A   | 0.76 (0.06) a, A   | 16.55 (0.53) cd, B  | 35.48 (1.13) b, A  | 11.58 (0.17) abc, B | 15.05 (0.13) b, A  |
| S - 5s           | 4.97 (0.04) e, B  | 5.71 (0.02) e, A   | 0.47 (0.03) e, A   | 0.27 (0.01) d, B   | 18.25 (0.21) ab, B  | 35.22 (1.48) b, A  | 12.59 (0.10) a, B   | 16.03 (0.08) a, A  |
| S - 10 s         | 8.13 (0.13) b, B  | 8.50 (0.06) a, A   | 0.49 (0.08) de, A  | 0.52 (0.02) c, A   | 15.59 (0.31) ed, B  | 41.00 (0.68) a, A  | 10.85 (0.25) cd, B  | 13.18 (0.25) ef, A |
| S - 15 s         | 10.12 (0.02) a, A | 7.17 (0.02) b, B   | 0.89 (0.06) a, A   | 0.61 (0.03) bc, B  | 15.51 (0.15) ed, B  | 40.13 (0.99) a, A  | 10.05 (0.21) d, B   | 13.78 (0.22) ed, A |

<sup>[a]</sup> CW = cold water, HW = hot water, S = steam. Values in the parentheses are standard deviations. The same lower-case letter in the same column for the specific type of sorghum indicate no significant difference ( $p \leq 0.05$ ). The same upper-case letter in the same row for a given treatment and density type indicate no significant difference.

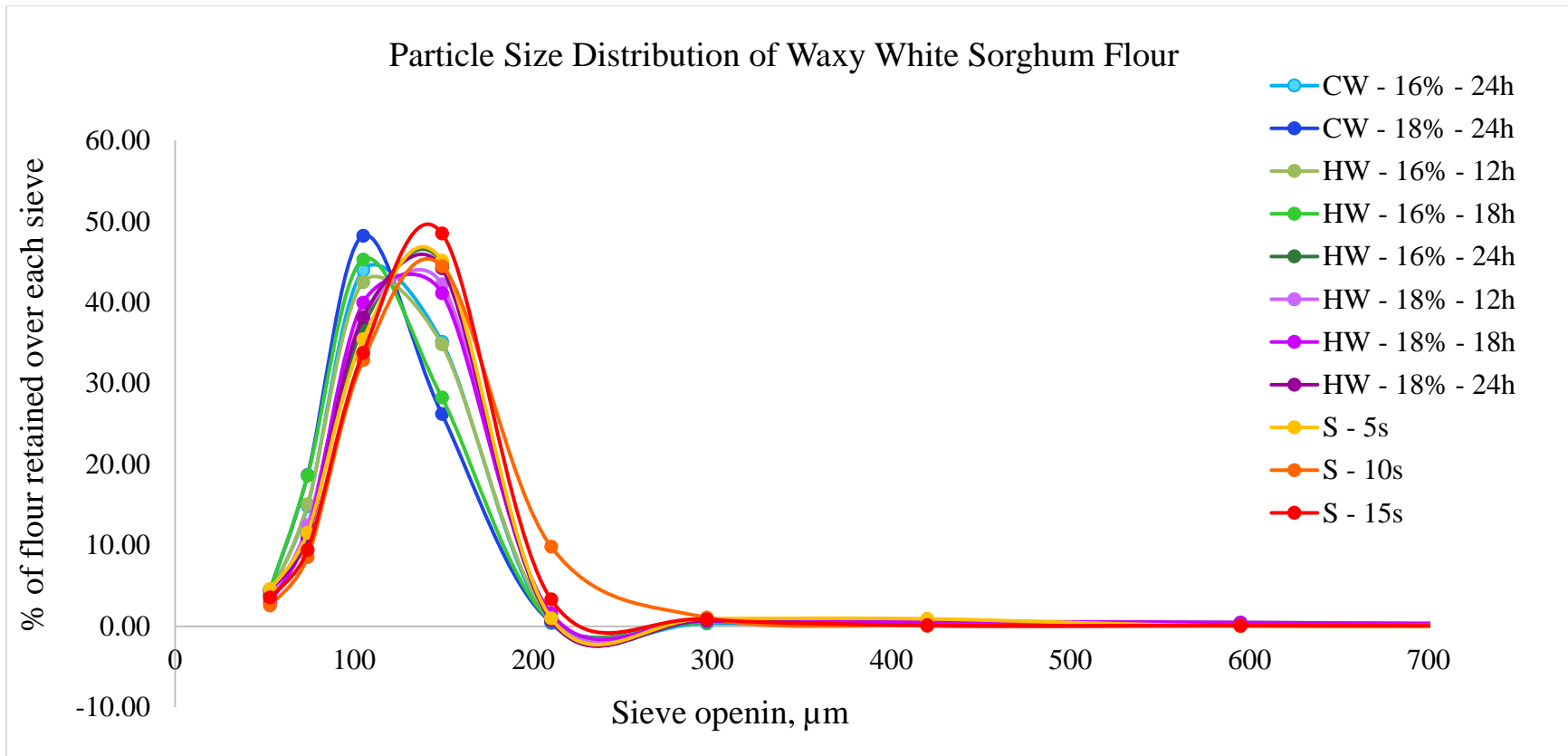
#### ***4.4.2. Particle Size Distribution of White and Waxy White Sorghum Flour***

The particle size distribution of the flour is dependent on the milling characteristics, tempering conditions and the kernel properties. From the particle size analysis, it was observed that more than 98% of the particles are below 212 $\mu$ m. This validates the claim to classify the milling fraction as flour as per the Code of Federal Regulations. Figures 3 and 4 represent the particle size distribution curve of the procured white and waxy white sorghum flour, respectively. From Fig 3 (white sorghum) it is evident that the percentage of flour below 105  $\mu$ m is decreased with increasing tempering moisture content when treated with cold water. However, the contrast was observed for waxy white sorghum flour (Fig.4). In case of hot water tempering, varying the tempering time or moisture content had not made any observable trend in the particle size distribution. Steam tempering to 10s and 15s produced less amount of flour  $\leq$  105  $\mu$ m than other treatments. Changing the tempering condition from cold water to steam reduced the production fine grained flour ( $\leq$  105  $\mu$ m).



\*Fig 4- 2. Particle Size Distribution of White Sorghum Flour

\*CW: Cold Water, HW: Hot Water, S: Steam



\*Fig 4- 3. Particle Size Distribution of Waxy White Sorghum Flour

\*CW: Cold Water, HW: Hot Water, S: Steam

### ***4.4.3. Proximate Composition of white and waxy white sorghum flour***

#### ***4.4.3.1. Total Starch***

The total starch of white and waxy white sorghum flour from all treatments was (81.18 – 88.20) % and (82.38 – 87.76) %. There was no significant difference ( $p < 0.05$ ) in total starch content of white and waxy white sorghum flour. Hot, cold or steam tempering had negligible impact on the total starch content (Table 4-4). Tempering moisture content and time did not influence the total starch content of both white and waxy white sorghum flour (Table. 4-4.). Similar results were reported by Zhao and Ambrose (2017) on studying red sorghum flour. The authors could not show any specific trend with increasing time of hot water tempering or type of tempering (cold water, hot water and steam) on the total starch content. Though there are several researches on greater presence of amylopectin in waxy sorghum than non-waxy sorghum varieties (Rooney and Serna-Saldivar 2000; Sang et al., 2008; Zhu, 2014), it is unsure if the total starch content in waxy sorghum is more than non-waxy varieties.

#### ***4.4.3.2. Ash***

According to Serna-Saldivar and Rooney (1995), the germ, pericarp and endosperm of sorghum kernel contains approximately 9.2%, 3.4% and 0.6% of ash, respectively. Hence, ash content determination in any particular flour is comparatively an accurate indicator of the separation of endosperm from pericarp and germ (Kent, 1975) than the other contamination detection methods in flour. White and waxy white sorghum grain kernel contained 1.67% and 2.20% of ash, respectively (Table. 4-6). When both the sorghum kernels were treated with cold water for 24h to bring to final m.c. of 18% (w.b.), the flour produced from the developed milling

flowsheet displayed the lowest ash content (0.63% for white sorghum flour and 0.94% for waxy white sorghum flour). Steam tempering was also efficient in producing flour with less ash. The heat and moisture combination during steam tempering toughened the bran and softened the endosperm. This might have resulted in the lower accumulation of bran components and thereby ash content in the flour compared to other tempering processes. The ash content in both the types of sorghum flours from all tempering treatments were significantly higher than reported by Zhao and Ambrose (2017) on roller milling red sorghum (0.33% to 0.43%), although lower than reported by Liu et al. (2012) (1.25% to 1.41%) on application of ADHM on white and red sorghum. This suggests that roller milling is a better milling technique to reduce ash content in flour. Tempering time, tempering moisture content or type of tempering did not impose any effect on the ash content of white and waxy white sorghum flour. Hot water and steam tempering time did not affect ash content of red sorghum flour (Zhao and Ambrose, 2017).

#### ***4.4.3.3. Protein***

Whole untempered waxy white sorghum kernel had greater protein content than whole untempered white sorghum kernel (Table. 4-6). Thus, all waxy white sorghum flour displayed the same trend when compared to white sorghum flour. A previous research indicated that the total protein content of waxy sorghum varieties is higher and is more evenly distributed in the proteinaceous matrix and bodies of the endosperm when compared to non-waxy varieties (Sullins and Rooney, 1975). The protein content of white sorghum flour and waxy white sorghum flour obtained after tempering treatments ranged from 7.61% - 8.78% and 8.58% - 10.17%, respectively (Table 4-5). The protein content was greater than reported for red sorghum flour containing 4.94% - 5.96% (Zhao and Ambrose, 2017) but less than the protein content in white sorghum flour (9.64% – 10.65%) reported by Liu et al. (2012). Tempering moisture content or time did not affect the

protein content of white and waxy white sorghum flour. Similar results were reported by Bai et al. (2018) on studying the effect of tempering moisture on protein composition of desi chickpea and barley flour. But steam tempering for 10 and 15s invariably reduced the protein content of the sorghum flour. The protein content of sorghum kernel was reported to be greater in the corneous endosperm when compared to the flourey endosperm (Ioerger et al., 2007). The lower protein content of white and waxy white sorghum flour produced from steam tempered kernels for 10s and 15s indicated that these tempering conditions had caused major portion of the corneous endosperm to be lost during milling when compared to the other applied tempering conditions which consequently reduced the protein content of the flour. The study by Blessing and Gregory (2010) on dehulled and undehulled mungbean flour reported heat treatment on mung beans reduces crude protein.

#### ***4.4.3.4. Crude Fiber***

The major insoluble fiber component of sorghum is cellulose which varies from 1.19 to 5.23% in sorghum varieties (Kamath and Belavady, 1980). Sorghum bran also contains insoluble dietary fiber which is reported to be reduced during milling (Serna-Saldivar, 1995; Kulamarva et al., 2009; Koreissi-Dembélé et al., 2013). This result aligned with the results obtained from this study where a reduction was observed in crude fiber of white and waxy white sorghum flours (Table. 4-5) when compared to their respective untempered kernels (Table. 4-6.). Both white and waxy white sorghum flour displayed no increasing or decreasing pattern in crude fiber content with tempering moisture content or time when tempered with cold water, hot water or steam.

#### **4.4.3.5. Crude Fat**

Results from Table 4-5 exhibit no specific trend in crude fat content of white and waxy white sorghum flour with hot water tempering, moisture content and time. Ineffectiveness of the same on crude fat content was also observed in red sorghum flour (Zhao and Ambrose, 2017). However, both types of sorghum flours showed a decrease in crude fat content with increasing moisture content during cold-water tempering and steam tempering (15s). This could be associated with increasing bran yield during milling of white and waxy white sorghum tempered at the mentioned conditions. As lipids in sorghum are located in the scutellum of the germ and aleurone layer (Rooney, 1978; Kulamarva et al., 2009) of the kernel, those will be removed during milling. Lipids in sorghum flour was reported to reduce significantly due to bran removal (Kebakile et al., 2008) and its composition was reported to be changed during milling and size reduction (Buitimea-Cantúa et al., 2013). Waxy white sorghum flour depicted to have more crude fat than white sorghum flour. This should probably be due to the high crude fat content in whole waxy white sorghum grain kernel (Table. 4-6).

#### **4.4.4. Damaged Starch**

Damaged starch in milled flour increases the water holding capacity of flour by increasing the water absorption. Higher concentration of damaged starch also increases the starch digestibility during the dough making due to higher levels of starch exposed for hydration and enzymatic action (Dendy and Dobraszczyk, 2001). Cold water tempering produced white sorghum flour with the least damaged starch. Damaged starch in white sorghum flour did not show any significant trend with increasing tempering moisture content or time period when treated with cold and hot water (Table 4-4) However, steam tempering to 10 and 15s produced white sorghum flour with the



greatest damaged starch content. All tempering treatments produced similar amount of damaged starch in waxy white sorghum flour. Thus, the implemented tempering conditions did not produce any effect on waxy white sorghum. Waxy white sorghum flour displayed greater damaged starch content when compared to white sorghum flour for the same treatment. This aligns with a study from a previous research where waxy sorghum starches were proven to be more susceptible to damage during milling than non-waxy starches (Zhu, 2014; Han et al., 2008) due to presence of more cracks and holes in waxy white sorghum kernel than the other (Yan et al., 2011). Previous research has indicated that hardness index of kernel and damaged starch content of flour are highly correlated (Barrera et al., 2007; Ghodke et al., 2009). This explains the reason of waxy white sorghum flour displaying greater starch damage when compared to white sorghum flour produced from most tempering conditions (Comparing Table 3-2 for hardness index and Table 4-4 for damaged starch content).

Table 4- 4. Total starch and damaged starch of white and waxy white sorghum flour <sup>[b]</sup>

| Treatment        | Total Starch (%)    |                    | Damaged starch (%) |                   |
|------------------|---------------------|--------------------|--------------------|-------------------|
|                  | White               | Waxy White         | White              | Waxy White        |
|                  | Sorghum             | Sorghum            | Sorghum            | Sorghum           |
| CW - 16 % - 24 h | 82.91 (2.42) bc, A  | 82.38 (1.48) b, A  | 5.49 (0.21) de, B  | 6.52 (0.12) b, A  |
| CW - 18 % - 24 h | 81.38 (1.53) c, A   | 82.53 (0.93) b, A  | 5.25 (0.06) e, B   | 7.96 (0.01) a, A  |
| HW - 16 % - 12h  | 81.18 (1.79) c, A   | 85.09 (2.26) ab, A | 6.65 (0.31) b, B   | 7.74 (0.12) a, A  |
| HW - 16 % - 18 h | 84.81 (1.75) abc, A | 85.01 (1.28) ab, A | 5.80 (0.21) cd, B  | 7.80 (0.05) a, A  |
| HW - 16 % - 24 h | 84.76 (1.76) abc, A | 86.34 (1.04) ab, A | 7.38 (0.22) a, A   | 6.90 (0.47) b, A  |
| HW - 18 % - 12 h | 85.13 (0.46) abc, B | 87.22 (0.80) a, A  | 6.71 (0.12) b, B   | 7.81 (0.25) a, A  |
| HW - 18 % - 18 h | 85.25 (1.50) abc, A | 86.79 (1.24) a, A  | 7.10 (0.07) ab, B  | 7.83 (0.09) a, A  |
| HW - 18 % - 24 h | 87.23 (1.93) ab, A  | 86.93 (2.27) a, A  | 6.16 (0.07) c, B   | 7.84 (0.05) a, A  |
| S - 5s           | 86.69 (2.37) ab, A  | 86.23 (1.00) ab, A | 6.02 (0.09) c, B   | 7.34 (0.29) ab, A |
| S - 10 s         | 86.99 (1.76) ab, A  | 87.02 (1.72) a, A  | 7.08 (0.01) ab, B  | 7.27 (0.05) ab, A |
| S - 15 s         | 88.20 (1.18) a, A   | 87.76 (1.27) a, A  | 7.35 (0.08) a, A   | 7.16 (0.59) ab, A |

<sup>[b]</sup> CW = cold water, HW = hot water, S = steam. Values in the parentheses are standard deviations. The same lower-case letter in the same column for the specific type of sorghum indicate no significant difference ( $p \leq 0.05$ ). The same upper-case letter in the same row for a given treatment and density type indicate no significant difference.

Table 4- 5. Proximate composition of milled white and waxy white sorghum flour <sup>[c]</sup>

| Treatment        | Protein (%)        |                   | Ash (%)           |                   | Crude Fat (%)     |                   | Crude Fiber (%)   |                    |
|------------------|--------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|--------------------|
|                  | White              | Waxy White        | White             | Waxy White        | White             | Waxy White        | White             | Waxy White         |
|                  | Sorghum            | Sorghum           | Sorghum           | Sorghum           | Sorghum           | Sorghum           | Sorghum           | Sorghum            |
| CW - 16 % - 24 h | 8.66 (0.06) abc, B | 10.17 (0.10) a, A | 0.95 (0.02) bc, B | 1.46 (0.04) bc, A | 0.82 (0.05) d, B  | 2.50 (0.05) cd, A | 0.56 (0.01) a, A  | 0.61 (0.02) de, A  |
| CW - 18 % - 24 h | 8.55 (0.17) abc, B | 9.72 (0.28) ab, A | 0.63 (0.04) e, B  | 0.94 (0.04) h, A  | 0.42 (0.05) e, B  | 0.72 (0.04) h, A  | 0.32 (0.04) d, A  | 0.46 (0.01) f, A   |
| HW - 16 % - 12 h | 8.72 (0.12) ab, B  | 9.88 (0.09) ab, A | 0.92 (0.04) c, B  | 1.11 (0.01) g, A  | 1.06 (0.01) c, B  | 0.82 (0.03) h, A  | 0.44 (0.05) bc, B | 0.73 (0.04) bc, A  |
| HW - 16 % - 18 h | 8.78 (0.11) a, B   | 10.08 (0.30) a, A | 1.02 (0.03) ab, B | 1.44 (0.03) c, A  | 1.98 (0.01) a, A  | 1.07 (0.03) g, B  | 0.55 (0.01) a, B  | 0.93 (0.04) a, A   |
| HW - 16 % - 24 h | 8.49 (0.20) abc, B | 10.11 (0.23) a, A | 0.93 (0.04) bc, B | 1.84 (0.02) a, A  | 1.70 (0.04) b, B  | 2.21 (0.01) e, A  | 0.50 (0.03) ab, B | 0.77 (0.05) bc, A  |
| HW - 18 % - 12 h | 8.35 (0.09) c, B   | 9.72 (0.14) ab, A | 0.87 (0.04) c, B  | 1.47 (0.04) ef, A | 1.83 (0.06) b, A  | 1.99 (0.05) f, A  | 0.32 (0.01) d, B  | 0.68 (0.04) bcd, A |
| HW - 18 % - 18 h | 8.39 (0.14) bc, B  | 9.87 (0.09) ab, A | 1.04 (0.04) a, B  | 1.46 (0.01) bc, A | 1.78 (0.07) b, B  | 2.96 (0.04) b, A  | 0.55 (0.02) a, B  | 0.78 (0.01) b, A   |
| HW - 18 % - 24 h | 8.36 (0.04) c, B   | 9.50 (0.10) b, A  | 0.94 (0.03) bc, B | 1.55 (0.04) b, A  | 1.10 (0.06) c, B  | 3.16 (0.05) a, A  | 0.59 (0.04) a, A  | 0.67 (0.05) cd, A  |
| S - 5s           | 8.45 (0.10) abc, B | 9.94 (0.11) ab, A | 0.75 (0.01) d, B  | 1.38 (0.04) cd, A | 0.36 (0.04) ef, B | 2.37 (0.04) d, A  | 0.38 (0.02) cd, B | 0.52 (0.03) ef, A  |
| S - 10 s         | 7.83 (0.16) d, B   | 8.60 (0.25) c, A  | 0.70 (0.01) de, B | 1.32 (0.02) de, A | 0.23 (0.05) f, B  | 2.56 (0.05) c, A  | 0.33 (0.05) d, B  | 0.49 (0.02) f, A   |
| S - 15 s         | 7.61 (0.09) d, B   | 8.58 (0.15) c, A  | 0.66 (0.02) e, B  | 1.16 (0.00) fg, A | 0.20 (0.04) f, B  | 1.96 (0.04) f, A  | 0.39 (0.04) cd, B | 0.71 (0.01) bcd, A |

<sup>[c]</sup> CW = cold water, HW = hot water, S = steam. Values in the parentheses are standard deviations. The same lower-case letter in the same column for the specific type of sorghum indicate no significant difference ( $p \leq 0.05$ ). The same upper-case letter in the same row for a given treatment and density type indicate no significant difference.

Table 4- 6. Proximate composition of untempered white and waxy white sorghum kernel

| Composition  | Untempered White<br>Sorghum Kernel<br>(w/w %) | Untempered Waxy White<br>Sorghum Kernel<br>(w/w %) |
|--------------|---|--|
| Total Starch | 59.64 (0.79)                                  | 71.35 (1.88)                                       |
| Protein      | 11.34 (0.12)                                  | 13.51 (0.01)                                       |
| Ash          | 1.61 (0.02)                                   | 2.20 (0.04)  |
| Crude Fiber  | 2.30 (0.00)                                   | 2.11 (0.05)  |
| Crude Fat    | 1.61 (0.02)                                   | 2.51 (0.19)  |

#### ***4.4.5. Moisture content of flour***

An increasing trend in moisture content of white sorghum flour was observed with increasing tempering moisture content when treated with cold and hot water (Table. 5.). The result aligns with the results obtained in a study by Patwa et al. (2014) where the moisture content of Hard Red Winter, Hard Red Spring and Soft Red Winter wheat flours raised with tempering moisture contents 12%, 14% and 16% w.b. Hot water tempering time did not produce any effect on the moisture content of flour, although steam tempering time significantly increased the moisture content of white sorghum flour.

A clear increase in moisture content of waxy white sorghum flour was observed with increasing tempering moisture content when treated with cold water. Steam tempering also increased the moisture content of flour with increasing tempering time. However, hot water tempering did not display any significant effect on the same. Thus, the indirect tempering of waxy white sorghum in a beaker placed in hot water at 60° C was inefficient in penetrating the moisture inside the waxy white sorghum kernel.

White and waxy white sorghum steam tempered to 15s produced flour with the highest moisture content (17.72% and 16.01% w.b. respectively). This can be attributed to the highest moisture content of the kernels tempered using steam for 15s (For all treatments, white sorghum flour displayed greater moisture content when compared to waxy white sorghum flour. This might be because of better permeability of the pericarp of white sorghum kernels.

#### ***4.4.6. Bulk, tapped and true density***

Bulk density of flour plays an important role in developing packaging material. Lower density flours occupy greater space consequently increasing packaging material per unit weight. Tapped density is expressed as compacted bulk density. True density is the density of the material with respect to the actual volume occupied by it excluding closed and open pores. The bulk, tapped and true density of white sorghum flour increased with increasing tempering moisture content when tempered with cold and hot water (Table 4-7). Similar results were found by Subramanian and Viswanathan (2007) on millet flours, Bengal gram flour (Raigar and Mishra, 2015) and chestnut flour (Zhu et al., 2012). Tempering time did not show any effect on the densities of white sorghum flour except when tempered with steam. This is due to insignificant change in moisture content of flour with increasing hot water tempering time (Table 4-7). The increment in the densities with tempering moisture content is attributed to the increasing cohesive forces between flour particles with increasing moisture content. Bulk density of flour can also be attributed to flour particle shape which could cause the flour particles to compact together. The inclusion of water during tempering caused the endosperm particles in the flour to expand in which its mass expansion was greater than its volume expansion increasing the density of flour (Patwa et al., 2014)). Steam tempering white sorghum to 15s produced flour with the highest bulk (484.06 kg/m<sup>3</sup>), tapped (581.40 kg/m<sup>3</sup>) and true (1478.83 kg/m<sup>3</sup>) density when compared to other 10 treatments.

Waxy white sorghum displayed no distinct tendency between the densities and tempering moisture content. The moisture content of waxy white sorghum grain flour obtained from grain tempered to final m.c. 18% w.b. with cold water for 24 hours and steam for 15s is 14.06% and 16.01 % w.b. respectively. Though the moisture content of waxy white sorghum flour showed an

increment in these tempering treatments, the bulk ( $427.33 \text{ kg/m}^3$  and  $396.41 \text{ kg/m}^3$ ), tapped ( $533.62 \text{ kg/m}^3$  and  $506.92 \text{ kg/m}^3$ ) and true ( $1453.70 \text{ kg/m}^3$  and  $1446.15 \text{ kg/m}^3$ ) densities showed a downward direction for the same treatments. Similar decrease in bulk and tapped density was observed in Hard Red Spring and Soft Red Winter wheat flours with increasing tempering moisture content in a study by Patwa et al. (2014).

The bulk, tapped and true densities of white sorghum flour was greater than the respective densities of waxy white sorghum flour from all treatments. This could be related to the moisture contents of the flour. Every applied tempering treatment to both types of sorghum produced white sorghum flour with greater moisture content than waxy white sorghum flour from the same tempering treatment. Thus, the endosperm expansion by mass was greater in white sorghum flour than waxy white sorghum flour corresponding to their respective volume expansion due to better moisture penetration in the former than the latter.

Table 4- 7. Moisture content and Density values of white and waxy white sorghum flour <sup>[d]</sup>

| Treatment        | Moisture Content (% w.b.) |                          | Bulk Density (kg/m <sup>3</sup> ) |                          | Tapped Density (kg/m <sup>3</sup> ) |                          | True Density (kg/m <sup>3</sup> ) |                          |
|------------------|---------------------------|--------------------------|-----------------------------------|--------------------------|-------------------------------------|--------------------------|-----------------------------------|--------------------------|
|                  | White Sorghum Flour       | Waxy White Sorghum Flour | White Sorghum Flour               | Waxy White Sorghum Flour | White Sorghum Flour                 | Waxy White Sorghum Flour | White Sorghum Flour               | Waxy White Sorghum Flour |
| CW - 16 % - 24 h | 13.08 (0.49) d, A         | 13.01 (0.10) d, A        | 451.90 (3.93) d, A                | 418.58 (2.48) ab, B      | 555.05 (5.71) c, A                  | 533.09 (4.28) a, B       | 1456.10 (0.87) d, A               | 1447.18 (2.66) b, B      |
| CW - 18 % - 24 h | 15.14 (0.52) bc, A        | 14.06 (0.40) b, B        | 467.15 (1.79) c, A                | 427.33 (4.60) a, B       | 567.30 (1.97) b, A                  | 533.62 (1.78) a, B       | 1464.72 (1.30) bc, A              | 1453.70 (3.16) a, B      |
| HW - 16 % - 12 h | 13.22 (0.10) d, A         | 12.80 (0.15) d, B        | 446.07 (2.03) e, A                | 405.87 (1.79) bcde, B    | 553.79 (3.68) c, A                  | 511.56 (1.78) bcd, B     | 1456.20 (0.57) d, A               | 1441.90 (2.26) bcd, B    |
| HW - 16 % - 18 h | 13.77 (0.03) d, A         | 12.97 (0.17) d, B        | 443.67 (1.95) e, A                | 413.56 (5.13) abc, B     | 556.09 (3.93) c, A                  | 520.09 (3.08) b, B       | 1454.83 (2.35) d, A               | 1441.97 (2.03) bcd, B    |
| HW - 16 % - 24 h | 13.78 (0.07) d, A         | 12.80 (0.23) d, B        | 455.00 (2.39) d, A                | 401.66 (11.87) cde, B    | 552.78 (4.03) c, A                  | 515.70 (5.16) bc, B      | 1457.55 (0.64) d, A               | 1438.23 (2.45) d, B      |
| HW - 18 % - 12 h | 14.68 (0.14) c, A         | 13.33 (0.20) cd, B       | 463.61 (1.73) c, A                | 411.83 (6.18) abcd, B    | 565.76 (0.55) b, A                  | 519.25 (5.43) b, B       | 1465.95 (2.47) bc, A              | 1437.53 (2.20) d, B      |
| HW - 18 % - 18 h | 15.20 (0.14) bc, A        | 13.31 (0.06) cd, B       | 466.34 (0.66) c, A                | 396.65 (1.27) de, B      | 567.11 (0.38) b, A                  | 503.87 (1.73) de, B      | 1462.20 (1.82) c, A               | 1440.77 (3.01) bcd, B    |
| HW - 18 % - 24 h | 15.49 (0.03) b, A         | 14.09 (0.03) b, B        | 469.03 (0.44) c, A                | 395.23 (4.34) de, B      | 568.32 (0.09) b, A                  | 501.42 (1.83) de, B      | 1463.80 (1.14) bc, A              | 1442.20 (0.57) bcd, B    |
| S - 5s           | 13.78 (0.23) d, A         | 12.04 (0.25) d, B        | 456.88 (1.64) d, A                | 406.28 (3.93) bcde, B    | 567.86 (4.05) b, A                  | 518.69 (4.29) b, B       | 1467.95 (2.90) b, A               | 1442.25 (1.11) bcd, B    |
| S - 10 s         | 15.37 (0.42) bc, A        | 13.56 (0.26) c, B        | 478.62 (1.63) b, A                | 394.54 (3.10) e, B       | 577.06 (1.79) a, A                  | 497.69 (4.18) e, B       | 1475.50 (0.53) a, A               | 1439.70 (1.60) cd, B     |
| S - 15 s         | 17.72 (0.19) a, A         | 16.01 (0.07) a, B        | 484.06 (0.72) a, A                | 396.41 (3.09) de, B      | 581.40 (1.95) a, A                  | 506.92 (4.02) cde, B     | 1478.83 (1.27) a, A               | 1446.15 (3.15) bc, B     |

<sup>[d]</sup> CW = cold water, HW = hot water, S = steam. Values in the parentheses are standard deviations. The same lower-case letter in the same column for the specific type of sorghum indicate no significant difference ( $p \leq 0.05$ ). The same upper-case letter in the same row for a given treatment and density type indicate no significant difference.



#### **4.4.7. Color analysis of flour**

##### **4.4.7.1. Lightness value**

The lightness value of flour could be directly related to the bran contamination in flour. Along with the ash content, Kim and Flores (1999) used color and bran speck counts to determine the bran contamination in wheat flours. White and waxy white sorghum flour depicted increment in lightness value of flour when tempering condition changed from cold water to steam. Steam tempered (for 10s and 15s) white and waxy white sorghum produced flour with the greatest lightness value (or brightest color) due to efficient separation of bran from endosperm when compared to other tempering conditions. This is also supported from the final bran quantity obtained by milling steam tempered (10s and 15s) white sorghum using the developed flowsheet (Table. 2). Tempering time and tempering moisture content did not seem to influence lightness value of white sorghum flour.

White sorghum flour displayed greater lightness value when compared to waxy white sorghum flour produced from the same tempering treatment. This could be related to the ash content in the flour (Table. 3). The ash content in waxy white sorghum flour was greater than white sorghum flour obtained from the same tempering treatment which indicated greater contamination in the former compared to the latter. This consequently decreased the lightness value of waxy white sorghum flour when compared to white sorghum flour.

##### **4.4.7.2. a-value**

The a-value measurement is based on a red to green comparison. The results from the table. 6 show that the a-value of both white and waxy white sorghum flour was inclined towards the red

side due to all positive a-values. However, waxy white sorghum flour displayed greater a-value than the other produced from every tempering treatment. The presence of bran and ash (which has a reddish-brown color) in the flour could result a high a-value. Thus, the greater bran and ash contamination of waxy white sorghum flour could have resulted in its higher a-value. White and waxy white sorghum flour produced from steam tempering for 15s displayed the lowest a-value due to least contamination of bran. This result also aligns with the greatest lightness value of the flour obtained from the same treatment.

#### **4.4.7.3. b-value**

The b-value ranges from the yellow to blue scale. The positive values for both white and waxy white sorghum flour show that it falls in the yellow range (Table. 6). The yellow color of flour is due to the presence of carotenoids in the proteinaceous matrix of endosperm (Aboubacar and Hamaker, 1999). Waxy white sorghum flour displayed greater b-value than white sorghum flour from all treatments. This could probably be due to greater amount of carotenoids in waxy white sorghum. Previous research on assessing carotenoid bioaccessibility from matured yellow endosperm sorghum varieties, decorticated yellow-endosperm sorghum bran fractions contained the highest carotenoid levels (9.87–13.70 mg/kg), while the lowest level (2.90–7.36 mg/kg) was found in the decorticated flour (Kean et al., 2011). This explains how steam tempering white and waxy white sorghum to 10s and 15s produced flour with the lowest b-value when compared to flour from all other treatments due to maximum amount of bran removed from the grain when tempered with the above two treatments. Cold and hot water tempering moisture content and time did not provide any significant influence on the b-value of both types of sorghum flour.

Table 4- 8. Color values of white and waxy white sorghum flour <sup>[e]</sup>

| Treatment        | L (lightness)       |                     | a (green to red)  |                   | b (blue to yellow) |                   |
|------------------|---------------------|---------------------|-------------------|-------------------|--------------------|-------------------|
|                  | White               | Waxy White          | White             | Waxy White        | White              | Waxy White        |
|                  | Sorghum Flour       | Sorghum Flour       | Sorghum Flour     | Sorghum Flour     | Sorghum Flour      | Sorghum Flour     |
| CW - 16 % - 24 h | 84.64 (0.29) e, A   | 79.83 (0.96) cd, B  | 0.32 (0.03) bc, B | 2.34 (0.09) b, A  | 10.15 (0.08) d, B  | 12.44 (0.12) b, A |
| CW - 18 % - 24 h | 86.08 (0.38) cd, A  | 80.67 (0.40) abc, B | 0.25 (0.03) d, B  | 2.12 (0.06) c, A  | 10.11 (0.08) d, B  | 12.26 (0.11) b, A |
| HW - 16 % - 12 h | 84.67 (0.00) e, A   | 80.22 (0.40) bc, B  | 0.29 (0.01) cd, B | 2.29 (0.05) b, A  | 10.70 (0.03) c, B  | 12.36 (0.18) b, A |
| HW - 16 % - 18 h | 84.78 (0.29) e, A   | 80.63 (0.58) abc, B | 0.38 (0.01) b, B  | 2.16 (0.04) c, A  | 11.17 (0.10) b, B  | 12.58 (0.13) b, A |
| HW - 16 % - 24 h | 85.69 (0.17) d, A   | 78.56 (0.05) d, B   | 0.33 (0.03) bc, B | 2.54 (0.08) a, A  | 11.69 (0.27) a, B  | 13.08 (0.22) a, A |
| HW - 18 % - 12 h | 86.55 (0.28) bc, A  | 80.26 (0.64) bc, B  | 0.35 (0.02) b, B  | 2.10 (0.09) c, A  | 10.93 (0.21) bc, B | 12.31 (0.06) b, A |
| HW - 18 % - 18 h | 84.67 (0.21) e, A   | 79.81 (0.82) cd, B  | 0.54 (0.04) a, B  | 2.10 (0.04) cd, A | 11.06 (0.07) b, B  | 12.45 (0.21) b, A |
| HW - 18 % - 24 h | 86.19 (0.07) bcd, A | 79.64 (0.40) cd, B  | 0.37 (0.04) b, B  | 2.16 (0.06) c, A  | 10.89 (0.02) bc, B | 12.64 (0.15) b, A |
| S - 5s           | 86.94 (0.26) ab, A  | 81.60 (0.48) ab, B  | 0.32 (0.03) bc, B | 2.07 (0.05) cd, A | 10.38 (0.08) d, B  | 12.41 (0.35) b, A |
| S - 10 s         | 87.68 (0.21) a, A   | 82.14 (0.21) a, B   | 0.15 (0.02) e, B  | 1.97 (0.05) d, A  | 9.40 (0.11) e, B   | 11.47 (0.07) c, A |
| S - 15 s         | 87.48 (0.20) a, A   | 82.09 (0.34) a, B   | 0.06 (0.01) f, B  | 1.72 (0.08) e, A  | 9.08 (0.03) f, B   | 11.65 (0.13) c, A |

<sup>[e]</sup> CW = cold water, HW = hot water, S = steam. Values in the parentheses are standard deviations. The same lower-case letter in the same column for the specific type of sorghum indicate no significant difference ( $p \leq 0.05$ ). The same upper-case letter in the same row for a given treatment and density type indicate no significant difference.

#### 4.5. Conclusion

The developed flowsheet using laboratory-scale roller mill produced an average of 60.24% flour from white sorghum. However, the same milling technique produced an average of 39.8% of flour yield from waxy white sorghum flour due to high production of shorts and flaking of endosperm. The bulk, tapped, and true density of white sorghum flour increased with increasing tempering moisture content and steam tempering time. However, the applied tempering methods did not affect the densities of waxy white sorghum flour. Hot water tempering time did not work efficiently on both white and waxy white sorghum. Steam tempering for 15s produced white and waxy white sorghum flour with greater total starch, least bran contamination, brightest color and low ash content. However, this tempering method produced the lowest flour yield and protein content and high damaged starch. Tempering white and waxy white sorghum with cold water for 24 hours to bring them to final m.c. 18% (w.b.) produced better flour yield without compromising the protein content of flour with the lowest ash content and damaged starch. The total starch content and color of flour from this method was lower than the flour produced from steam tempering for 15s. 98% of the total white and waxy white sorghum flour produced from all tempering methods was less than 212  $\mu\text{m}$  claiming to be flour under CFR. The developed milling flowsheet using laboratory-scale roller mill was proven to be more productive on white sorghum than waxy white sorghum. Tempering white and waxy white sorghum with cold water for 24 hours to final m.c. 18% (w.b.) could be a suitable tempering method to obtain good flour yield and flour characteristics. However, the scaling up, cost estimation and energy consumption assessment of the developed technique are to be evaluated.

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## **Chapter 5 EFFECTS OF TEMPERING ON WHITE AND WAXY WHITE SORGHUM BREAD PROPERTIES**

### **5.1. Introduction**

Celiac is an autoimmune disease caused by the ingestion of gluten from wheat, rye and barley which leads to severe damage of villi in the small intestine that affects the absorption of essential nutrients for the body (Green and Cellier, 2007; Fasano and Catassi, 2010). Besides celiac disease, there are other related disorders like non-celiac gluten/wheat sensitivity and wheat allergy that have been rapidly gaining exposure (Sapone et al., 2012). According to recent reports from the Celiac Disease Foundation (2018), the prevalence of celiac disease is approximately to 0.5% of the total population in Africa and North America, 0.6% in Asia, and 0.8% in Europe and Oceania, with the prevalence being relatively lower in male than female individuals and lower in adults than children. Celiac disease is known to have no perpetual treatment and the keystone remedy is to follow a gluten-free (GF) diet (Green and Cellier, 2007; Fasano and Catassi, 2010; Sapone et al., 2012). Previous research has also indicated that the number of people adopting a GF diet has been higher than the number of celiac patients (Sapone et al., 2012).

These are the key factors in the world-wide expansion of GF market. Grand View Research (2019) predicted the global GF products market to expand at a CAGR of 9.1% from 2019 to 2025. Shahbandeh (2019) announced USA to be one of the leading countries in launching new GF products after Brazil from 2008 to 2018.

Sorghum is a GF cereal and fifth most important cereal after maize, rice, wheat and barley. It is also a hardy crop which can tolerate drought and requires less water for growth (Rai et al., 2008). Hence it can be widely grown in regions adversely affected by climatic change and water

scarcity (Padulosi et al., 2009; Hadebe et al., 2017). Previous researches have positively associated the nutritional benefits of sorghum with certain cancers (Awika and Rooney, 2004), cardiovascular health (Awika and Rooney, 2004), human melanoma (Gomez-Cordoves et al., 2001) and cholesterol (Carr et al., 2005). The United States Department of Agriculture (USDA) (2019) assessed that the world sorghum production for the years 2019 - 2020 will approximately be 57.90 million metric tons with United States leading in sorghum production among all countries with an estimate of 9.08 million metric tons for the same time period. However, most sorghum produced in the US has been widely used for ethanol production and making animal feed (Wang et al., 2000). Sorghum has also been regarded safe for celiac patients and considering its several advantages, it would be a very suitable base for the gluten-free food market (Kasarda, 2001; Vallons et al., 2010).

Bread has been the staple food for many cultures and countries in the world for many years (Yano, 2019). Hence, it holds a crucial position in the bakery market. The bakery sector is reported to have the lead in gluten-free products market (Grand View Research, 2019; Reports and Data, 2019). Wheat flour (primarily used to make wheat bread) dough comprises of a complex network of gluten, starch and water which captures gas during fermentation allowing the dough to rise (Schober et al., 2005; Cornish et al., 2006). Besides this, gluten provides the dough a viscoelastic nature causing it to expand and extend due to its network-forming capability which helps retain the gas in the dough during fermentation and leavening providing the product a desirable porous structure (Schober et al., 2007). Gluten also plays an important role in moisture retention and shelf-life stability of the product (Cornish et al., 2006). Gluten-free cereals gives a “runny” cake-like batter due to its incapability of network formation (Cauvain, 1998). The absence of gluten would omit all the attributes necessary for a good bread. Thus, making gluten-free bread has been challenging for years.

Several attempts have been made in the past regarding making sorghum bread. To substitute the function of gluten, several ingredients have been used. Hydrocolloids like xanthan gum, guar gum, etc. have been used as a thickening agent to keep the starch granules, yeast and gas bubbles suspended during fermentation (Cauvain, 1998; Schober, 2009); starches like corn starch (Schober et al., 2005), rice starch (Onyango et al., 2011) or potato starch (Schober et al., 2007; Onyango et al., 2011) cause gelatinization to occur faster which facilitates development of a cohesive crumb that traps gas bubbles and prevents the crust from collapsing (Taylor et al., 2006) and, milk powder for a stronger structure (Cauvain, 1998; Schober et al., 2005; Schober et al., 2007).

Flour particle size has been reported to play a significant role on gluten-free bread quality (De La Hera et al., 2014;) which is depended to a great extent on the milling technique performed. The other factors which affect the quality of bread are ash content of flour which also depends on bran separation during milling, and water absorption capacity of flour during dough formation which is affected by the damaged starch content in flour. These flour characteristics are affected by milling (Mousia et al., 2004; Lijuan et al., 2007) to a great extent due to the involvement of size reduction and separation of bran and germ from endosperm to procure flour. Previous research has indicated that roller milling was more efficient in extracting sorghum flour and good baking properties (Iva, 2014; Kebakile et al., 2007). The popularity of roller milling in the US also prevents the development of a separate milling technique for sorghum. Tempering is an important pre-milling conditioning to the grain and is the process of adding water to the grain to toughen the bran and mellow the endosperm to efficiently scrape out the bran and reduce the endosperm to flour. Kweon et al. (2009) in the study on effects of tempering conditions on milling and flour properties of soft wheat, proposed that tempering moisture content has a significant effect on bran

extraction. For sorghum, Zhao et al. (2018) concluded that steam tempering of sorghum at high pressure and temperature improved the separation of bran and endosperm without affecting the flour quality.

The following study hypothesizes that tempering can affect bread properties due to the effect of tempering on flour properties. Different tempering conditions were applied on white and waxy white sorghum which was milled using laboratory-scale table-top roller mill to procure white and waxy white sorghum flour. The objective of this study was to determine the effects of the applied tempering conditions on white and waxy white sorghum bread characteristics. This study also compares between white and waxy white sorghum bread characteristics for every tempering treatment applied on the grain.

## **5.2. Materials and Methods**

### ***5.2.1. White and Waxy White Sorghum Sample Preparation***

White and waxy white sorghum kernels were chosen for this study. 45.36 kg of each type of sorghum free from foreign matter such as dust, dirt, stones and chaff was procured from Nu Life Market, Scott City, Kansas for this study. The initial moisture content (m.c.) (wet basis (w.b.)) of white and waxy white sorghum kernels was measured using ASABE Standard S352.2 method (ASABE, 2012):

$$\text{moisture content (wet basis)} = \frac{m_w}{(m_w + m_d)} \times 100 \quad (1)$$

Where,  $m_w$  = the mass of water removed (g)

$m_d$  = the mass of sample after drying at 130° C for 18h (g)

The initial moisture content (w.b.) of white and waxy white sorghum kernels was found to be 13.46% and 13.29%, respectively.

The procured white and waxy white sorghum samples were conditioned using cold and hot water to bring them to final m.c. of 16 and 18% (w.b.) and with steam at 20 psi for 5, 10 and 15s. The samples were tempered with cold water for 24 hours and hot water at 60° C for 12, 18 and 24 hours.

*Cold water tempering:* White and waxy white sorghum samples were tempered with predetermined amount of cold distilled water (water available at room temperature) in a tempering drum to bring them to final m.c. of 16 and 18% (w.b.). According to AACC Official Method 26–95.01 (AACC, 1999), the amount of water to be added to the grain to bring it to the desired final m.c. is shown in equation 2. The tempering drums with the kernels and added water were rotated continuously for 20 min to allow the water to come in uniform contact with all the kernels. These moisture contents were chosen from previous research by Zhao and Ambrose, 2017 on red sorghum

$$Q = \frac{W(M_f - M_i)}{(100 - M_f)} \quad (2)$$

Where, Q = amount of distilled water required for tempering (ml)

W = the weight of sample required to be tempered (Kg)

M<sub>f</sub> = moisture content (% w.b.) to which the grain must be tempered

M<sub>i</sub> = initial moisture content (% w.b.) of sample

The cold water tempered samples were air tightly packed in zip lock bags. The tempered samples were held at room temperature for 24 h for uniform distribution of moisture throughout the samples and transferred to refrigerator at 5°C to maintain the moisture content of tempered samples.

*Hot water tempering:* White and waxy white sorghum were treated with calculated amount of distilled water in rotating tempering drums using equation (2) to bring them to final m.c. of 16 and 18% (w.b.). The treated samples were filled into glass beakers and sealed with aluminum foil to prevent water from evaporating. The beakers containing the treated samples were placed in hot water bath at 60° C for 12, 18, and 24 h. After tempering the samples for respective time periods, they were laid on trays to cool to room temperature and milled.

*Steam Tempering:* The samples were tempered with steam at 20 psi for 5, 10, and 15 sec in a hollow container with a screw barrel passing horizontally through the inside of the container to mix the samples as steam passed through the steam inlet on the lid of the container allowing uniform distribution of steam throughout the sample. After steam treatment, the samples were spread on flat trays to cool them to room temperature. The samples were transferred to ziplock bags and stored in refrigerator at 5° C to maintain the moisture content of the samples.

### ***5.2.2. Sorghum Roller Milling***

Both tempered white and waxy white sorghum was milled using laboratory-scale tabletop roller mill (Ross, Oklahoma City, Okla.). The milling flowchart (Fig. 4-1.) was designed after several milling trial methods on tempered white and waxy sorghum to obtain greater flour extraction with lesser bran contamination. The roller milling process consisted of four pairs of break rolls and eight pairs of reduction rolls, of which three pairs of reduction rolls were used for



milling coarse grits and five were used for milling fine grits. The milling outcomes from each pair of rolls was passed through a set of sifters (Fig. 4-1) and the milling stocks from each sieve was collected and weighed to separate the outcomes based on their size. The first two pairs of break rolls broke the kernel to smaller particles keeping the bran intact, and the last two scrapped the bran from the endosperm. Reduction rolls ground the endosperm grits to flour.

Coarse and fine grits were collected from each pair of break rolls based on their particle size and bran content. All collected coarse grits were first ground to flour by passing through the pair of smooth reduction rolls. The fine grits obtained from the coarse grit milling was collectively combined with the fine grits obtained from all the pairs of break rolls. The accumulated fine grits were passed through the smooth reduction rolls. Flour < 150µm and < 193µm was collected from all pairs of break rolls and reduction rolls respectively to calculate the amount of flour produced through this milling process. The milling process was performed in triplicates for every tempering treatment on white and waxy white sorghum. The milling fractions collected from the entire milling process were bran, fine bran, shorts, red dog and flour. The milling fractions were collected as shown in Fig. 4-1. at the end of the milling process from each replicate of a tempering treatment and combined to calculate the yield (%) of each fraction. The yield (%) of each milling fraction was calculated using equation (3)

$$Yield (\%) = \frac{\text{Weight of total milling fraction obtained from milling (g)}}{\text{Initial weight of grain taken (g)}} \times 100 \quad (3)$$

The break rolls were set to have a speed differential of 2.5:1 with the roll disposition being dull-to-dull and speed differential of the reduction rolls were maintained at 1.25:1. From prior experiments, the roll gaps for both break and reduction rolls were fixed as shown in Fig. 4-1. The sieve sizes used for separating the milling outcomes are explained in Table 4-1.

### ***5.2.3. Flour Particle Size Analysis***

The particle size analysis of white and waxy white sorghum flour samples was determined according to ASABE Standard S319.4 (ASABE Standards, 2008). The sieves (U.S. series) used for this study were 6, 8, 12, 16, 20, 30, 40, 50, 70, 100, 140, 200, 270, and pan. The empty weight of each sieve was measured and arranged in a sieve shaker (Ro-Tap model RX-29, W.S. Tyler, Mentor, Ohio) in the order stated above. The apertures of the sieves in the stated order decreased successively from 4760  $\mu\text{m}$  to 53  $\mu\text{m}$  for US sieve number 6 to 270 respectively. Flour ( $100 \pm 0.1$  g) was poured into the topmost sieve and the sieve was shut with a lid to enclose the stack of sieves. To assist smooth flow of flour through the sieves, a set of cleaner with nylon brushes and ivory rubber balls (39 mm) were placed on the sieves. There was one cleaner with nylon brushes and one ivory rubber ball on each of the sieves numbered 40, 50 and 70. Sieves with numbers 100, 140, 200 and 270 each had only one cleaner with nylon brushes on them. The sieve shaker shook for 10 min after which the mass of flour on each sieve was recorded. The particle size distribution curve for both white and waxy white sorghum flour was plotted.

### ***5.2.4. Bread Formulation***

Three loaves of bread were made from each tempering treatment on white and waxy white sorghum. The following formulation was used to make white and waxy white sorghum bread with all ingredients in flour weight basis (fwb) (%): Milled sorghum (white and waxy white sorghum) flour (90%), white rice flour (Bob's Red Mill Natural Foods, Inc. Milwaukee, Oregon, USA ) (10%), refine white sugar (6%), emulsified shortening (5%) (Sweetex, Stratas Foods, Memphis, Tennessee, USA), xanthan gum (3%) (Judee's Gluten Free, Columbus, Ohio, USA), double acting

baking powder (Monocalcium phosphate and sodium aluminium sulfate) (8%), common salt (1.5%) and distilled water (110%).

The batter was made in KitchenAid 6 Quart Professional 600 mixer with a coated flat beater (Whirlpool Cooperation, Benton Charter Township, Michigan, USA). The shortening and dry ingredients were weighed and first added to the mixing bowl after which the required amount of distilled water was added to the same. All ingredients were mixed with the flat beater paddle on speed 1 for 30 s. The batter was scraped down with a rubber spatula and mixed on speed 2 for 1.5 min. The final batter was weighed and transferred into greased metal pup loaf baking pans (15x9x5 cm<sup>3</sup>) and baked in a rotary baking oven (Reed Oven Co., Kansas City, Missouri) at 204° C for 30 min. Loaves were withdrawn immediately from the pans after removal from the oven and cooled for 2 hours on wire racks to bring to room temperature. After cooling the breads, they were weighed to evaluate the loaf weight and bagged individually in polyethylene bags. The packed loaves were stored at room temperature overnight.

#### ***5.2.5. Bread Characteristics***

Volume index, internal crumb analysis and crumb texture were analyzed 24 hours after baking.

##### ***5.2.5.1. Bake Loss***

Bake loss defined as the amount of water and organic material (sugars fermented and released as CO<sub>2</sub>) lost during baking (Alvarez-Jubete et al., 2010). The bake loss was calculated right after the loaves cooled down to room temperature and before packing them into polyethylene bags. The batter weight was evaluated for the batter from each tempering treatment on white and

waxy white sorghum before baking it to bread and the bread loaves made from the same treatment was weighed after removing them from the oven and cooling to room temperature. The bake loss for each loaf was evaluated using equation 4.

$$\text{Bake loss (g)} = (\text{Batter weight})(g) - (\text{Loaf weight})(g) \quad (4)$$

**5.2.5.2. Volume Index**

A single 3 cm wide slice was cut along the long dimension from the center of each loaf using a slice regulator. Volume index was determined using a cake template as described in AACC International Approved Method 10-91.01 (Fig. 5-2). Arin Akin and Miller (2017) measured the volume index of chemically leavened sorghum bread using the same technique. The cut slice was placed vertically against the template and the volume index was calculated using the following equation 5:

$$\text{Volume Index (mm)} = B + C + D \quad (5)$$

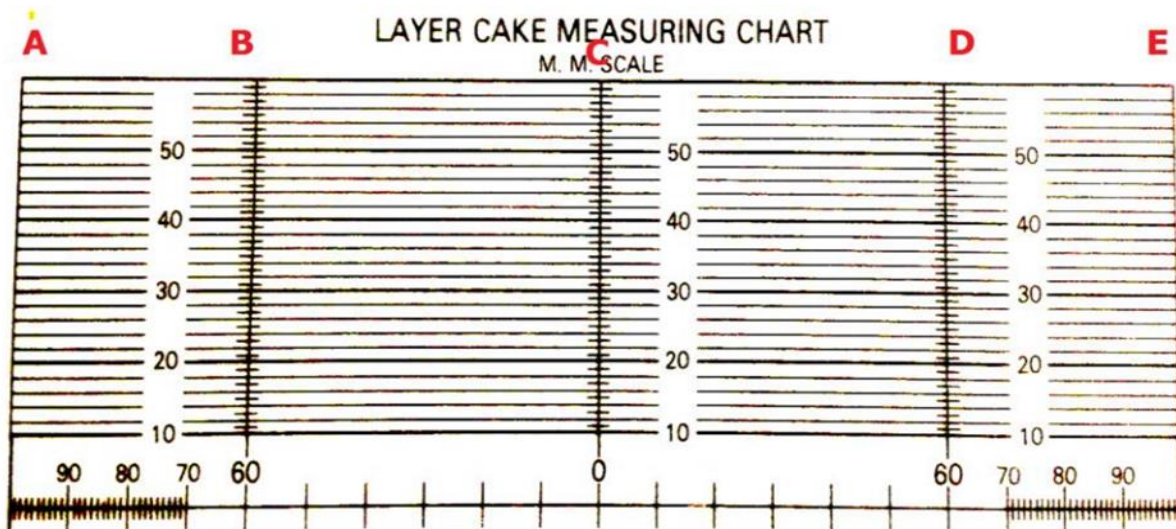


Fig 5- 1. Layer cake measuring chart for calculating volume index

### **5.2.5.3. Internal Crumb Structure**

The internal crumb structure of both white and waxy white sorghum bread was evaluated using C-Cell (Calibre Control International Ltd., Appleton, Warrington, United Kingdom) according to AACCC Method 10-18.01 Measurement of Crumb Structure of Baked Products by C-Cell. Image analysis software (C-Cell Software Version 2.0) was used to quantify the crumb cell characteristics. The features of interest for this study were cell number, cell diameter, cell wall thickness and slice brightness. For this analysis, one square slice of side 4 cm was cut off the crumb from the long slice of length 14 cm and width 3 cm used for evaluating the volume index of the bread loaf. The cut square crumb slice was scanned and photographed by the C-Cell imaging system to evaluate the crumb cell characteristics. Three square slices were analyzed for each treatment.

### **5.2.5.4. Texture Profile**

Resilience and Firmness of the baked white and waxy white sorghum bread loaves was evaluated using a TA.XT *Plus* Texture Analyzer (Texture Technologies Corp., Scarsdale, NY/Stable Micro Systems, Godalming, Surrey, UK) in accordance with AACCI Method 74-10.02 Measurement of Bread Firmness-Compression Test. The square crumb slice cut from each loaf was used immediately for crumb texture profile analysis after C-Cell analysis. The force applied to compress the center of the slice by the probe was set at 5.0 g. The maximum force required to compress the slice up to 40% of its thickness during the first compression is determined as the firmness of the crumb. The resilience measures the force with which the product tries to regain its original height after compression. It is measured after the withdrawal of the first compression and calculated as a percent of the upstroke force of the crumb applied to the downstroke force of the probe on the crumb.

### ***5.2.6. Statistical Analysis***

The study was a completely randomized design with 11 treatments each for white and waxy white sorghum. All experiments were conducted in triplicates for each treatment. The statistical significance between treatments on white sorghum and waxy white sorghum was analyzed separately, and between white and waxy white sorghum for the same treatment was also interpreted using PROC GLM in SAS (ver. 9.3, SAS Institute, Inc., Cary, N.C.). Based on ANOVA, significant differences ( $\alpha = 0.05$ ) between treatments was compared.

### **5.3. Results and Discussion**

The previous chapter explains the milling outcomes from all tempering treatments on both white and waxy white sorghum. Since bran plays an important role in determining the contamination in flour which effects baking, only bran extraction is reported in this chapter.

#### ***5.3.1. Milling outcomes***

##### ***5.3.1.1. Bran***

Bran extraction from the grain during milling was evidently increased with increasing moisture content when tempered with cold water, hot water or steam (Table. 4-2). Steam tempering for 10 and 15s provided the most efficient separation of bran from white and waxy white sorghum grain using the milling process. This consequently made the flour obtained from the treatments to have the least bran contamination. This aligns with the results from the previous research where Zhao (2016) concluded that steam tempering was most efficient in bran removal due to the combined effect of heat and pressure, which has caused the outer pericarp to easily scrape off during milling.

#### ***5.3.2. Particle Size Distribution of White and Waxy White Sorghum Flour***

Both white and waxy white sorghum flour produced from all tempering treatments had 98% of the total flour produced  $\leq 212 \mu\text{m}$ , which validates the requirement to be claimed as flour according to CFR. Figures 4-2 and 4-3 represent the particle size distribution curve of the procured white and waxy white sorghum flour respectively. The amount of white sorghum flour  $\leq 105 \mu\text{m}$  decreased with increasing tempering moisture content when treated with cold water. However, the contrast was observed for waxy white sorghum flour. There was no significant trend observed in

the amount of flour  $\leq 105 \mu\text{m}$  with hot water tempering time period or moisture content. Steam tempering to 10s and 15s produced less amount of flour  $\leq 105 \mu\text{m}$  than other treatments applied. Changing the tempering condition from cold water to steam reduced the production fine grained flour ( $\leq 105 \mu\text{m}$ ).

### **5.3.3. Bake loss**

Type of tempering, increasing moisture content or tempering time did not play any role or effect on the bake loss for both white and waxy white sorghum breads. For most treatments, waxy white sorghum bread displayed greater or same bake loss when compared to white sorghum bread from the same treatment (Table 5-1). Bake loss is an important bread characteristic due to its relationship with firming and staling of starch-based baked products (Miñarro et al., 2012). The presence of hydrocolloids like xanthan gum has helped to retain the water in bread which reduces bake loss (Alvarez-Jubete et al., 2010). The bake loss from this study was less than the bake loss in white and red sorghum breads (13.4-14.7%) from another study by Schober et al. (2005). However, the obtained bake loss was greater than bake loss in sorghum bread made from red sorghum flour and rice starch in the same ratio as in the current study (90:10) (7.2%) (Onyango et al., 2011).

### **5.3.4. Volume index**

Steam tempered and hot water tempered white sorghum displayed bread with the greatest (178.86 mm) and lowest volume index (163.92 mm) respectively. Tempering moisture content and tempering time displayed no effect on the volume index of both white and waxy white sorghum bread (Table 5-1). However, flour procured from white sorghum grain tempered with cold water



for 24 hours to bring to final m.c. 18% (w.b.) produced bread with high volume index. High volume index is an indication of good expansion in the bread and good quality. There was no difference observed between the volume index of white and waxy white sorghum bread from the same tempering treatment.

Bread volume is an important quality attribute of bread and depends on several factors like viscosity of the batter and the presence of surface-active components Schober et al. (2005).

Table 5- 1. Bake loss and volume index of white waxy white sorghum bread<sup>[a]</sup>

| Treatment        | Bake loss (%)      |                    | Volume Index (mm)    |                       |
|------------------|--------------------|--------------------|----------------------|-----------------------|
|                  | White Sorghum      | Waxy White Sorghum | White Sorghum        | Waxy White Sorghum    |
| CW - 16 % - 24 h | 9.51 (0.73) abc, B | 11.87 (0.12) a, A  | 167.00 (2.83) cd, A  | 167.00 (1.41) ab, A   |
| CW - 18 % - 24 h | 10.59 (0.57) ab, B | 11.87 (0.06) a, A  | 176.50 (2.12) ab, A  | 151.50 (2.12) e, B    |
| HW - 16 % - 12h  | 9.02 (0.66) bc, B  | 11.54 (0.89) ab, A | 163.00 (4.24) cde, A | 160.50 (3.54) abcd, A |
| HW - 16 % - 18 h | 10.65 (0.40) ab, A | 11.45 (0.45) ab, A | 165.00 (1.41) cde, A | 156.00 (2.83) de, A   |
| HW - 16 % - 24 h | 10.41 (0.29) ab, A | 10.97 (0.34) ab, A | 160.00 (2.83) de, A  | 155.67 (1.53) de, A   |
| HW - 18 % - 12 h | 10.72 (0.61) a, A  | 10.59 (0.32) b, A  | 165.50 (0.71) cde, A | 164.50 (0.71) abc, A  |
| HW - 18 % - 18 h | 10.22 (0.50) ab, B | 11.78 (0.32) a, A  | 156.50 (3.54) e, A   | 162.50 (2.12) abcd, A |
| HW - 18 % - 24 h | 8.31 (0.67) c, B   | 11.57 (0.30) ab, A | 170.33 (2.89) bc, A  | 151.00 (2.83) e, B    |
| S - 5s           | 9.12 (0.54) bc, B  | 11.23 (0.12) ab, A | 178.50 (2.12) ab, A  | 168.00 (2.83) a, A    |
| S - 10 s         | 9.80 (0.12) abc, B | 11.53 (0.19) ab, A | 178.67 (0.58) ab, A  | 160.00 (2.83) bcd, B  |
| S - 15 s         | 9.96 (0.50) ab, B  | 10.83 (0.15) ab, A | 179.50 (3.54) a, A   | 159.00 (1.00) cd, B   |

<sup>[a]</sup> CW = cold water, HW = hot water, S = steam. Values in the parentheses are standard deviations. The same lower-case letter in the same column for the specific type of sorghum indicate no significant difference ( $p \leq 0.05$ ). The same upper-case letter in the same row for a given treatment and density type indicate no significant difference.

### ***5.3.5. Internal crumb grain characteristics***

#### ***5.3.5.1. Slice Brightness***

The slice brightness of white sorghum bread decreased from 128.84 to 123.5 to 113.98 when the tempering treatment changed from steam to cold water to hot water respectively. While, waxy white sorghum bread produced from cold water and hot water tempering displayed no difference in slice brightness values. Steam tempered white and waxy white sorghum produced flour with the lowest bran contamination and ash content (Table 4-2 and Table 4-4). This has caused the bread produced from the same to have the brightest color. A strong negative correlation between slice brightness and ash content of flour ( $r = -0.84$ ) indicated that the slice brightness of the bread crumb was significantly influenced by the ash content in the flour. Thus, white and waxy white sorghum tempered with steam for 15s produced flour with lowest bran contamination and highest lightness value which consequently produced bread with the brightest slice (131.60 for white sorghum bread and 61.77 for waxy white sorghum bread) (Table 5-2). However, tempering time and moisture content did not affect the slice brightness of both white and waxy white sorghum bread. Slice brightness of white sorghum bread was greater than that of waxy white sorghum bread produced from the same tempering treatment. This can be related to the greater bran and ash contamination of waxy white sorghum flour and greater lightness value of white sorghum flour (Table 4-2, Table 4-4 and Table 4-7).

#### ***5.3.5.2. Number of Crumb Cells***

There has not been an observable relation between type of tempering and the average number of crumb cells in white sorghum bread (Table 5-2). However, hot water tempering of waxy

white sorghum produced bread with greatest average number of cells (1209.13) when compared to steam (1072.38) and cold-water tempering (1034.50). Tempering moisture content and tempering time did not provide any significant effect on average number of cells for both white and waxy white sorghum bread. White sorghum bread crumb appeared to have greater number of cells than waxy white sorghum bread from every tempering treatment. This could be attributed to the lesser amount of ash content and damaged starch content in white sorghum flour when compared to waxy white sorghum flour produced from all tempering conditions. In support to this statement, white sorghum flour displayed negative correlation between average number of crumb cells and damaged starch content ( $r = -0.21$ ), and between average number of crumb cells and ash content ( $r = -0.05$ ). Flour from white sorghum tempered with cold water for 24 hours to bring to final m.c. 18% (w.b.) and flour from white sorghum tempered with hot water for 18 hours to bring to final m.c. 16% (w.b.) produced bread with high number of crumb cells: 2051.00 and 1915.33 respectively. Similarly, waxy white sorghum tempered with cold water for 24 hours to bring to final m.c. 18% (w.b.) also produced bread with high number of crumb cells (1050.50).

#### ***5.3.5.3. Cell Diameter and Cell Wall thickness***

Similar to other crumb grain characteristics, change of tempering conditions from cold water to hot water or steam, tempering moisture content and tempering time did not display any effect on the cell diameter and cell wall thickness of both white and waxy white sorghum bread (Table 5-2). However, waxy white sorghum bread crumb possessed the greater average cell diameter (2.28 mm) and average cell wall thickness (0.45 mm) than white sorghum bread (1.04 mm and 0.36 mm respectively) for an individual tempering treatment.

According to Schober et al. (2005), fine crumb structure with greater number of crumb cells per unit area with smaller diameter is preferred. The same research also indicated that fine

crumb structure of sorghum bread would collaborate well with greater crumb firmness. This was unlike for white wheat pan bread where finer crumb structure is preferred along with lower firmness (Hoseney, 1998). The research by Schober et al. (2005), by proposed that greater starch damage strongly correlated with smaller number of cells and lower firmness. In this study, cell wall thick showed a positive correlation with damaged starch content ( $r = 0.16$ ) which supports the greater cell wall thickness in waxy white sorghum breads due to greater damaged starch content. Similar results were observed in this study where waxy white sorghum flour had greater damaged starch (Table 4-3) and the bread made from waxy white sorghum flour showed crumb structure with smaller diameter and greater softness. A positive correlation ( $r = 0.22$ ) was observed between damaged starch content of waxy white sorghum flour and cell diameter. The same study by Schober et al. (2005) also proposed that higher starch damage can be caused by greater kernel hardness. In a preliminary study to this study, the waxy white sorghum grain was reported to have greater mean kernel harness than white sorghum for all tempered and untempered conditions. This also supports the results of waxy white sorghum bread having low crumb quality. A study by Ahlborn et al. (2005) noted that crumb cells with larger diameters imply gas cell coalescence

Table 5- 2. C-cell analysis of white and waxy white sorghum bread <sup>[b]</sup>

| Treatment        | Slice Brightness     |                      | Number of cells         |                       | Cell wall thickness (mm) |                   | Cell wall diameter (mm) |                   |
|------------------|----------------------|----------------------|-------------------------|-----------------------|--------------------------|-------------------|-------------------------|-------------------|
|                  | White                | Waxy White           | White                   | Waxy White            | White                    | Waxy White        | White                   | Waxy White        |
|                  | Sorghum Bread        | Sorghum Bread        | Sorghum Bread           | Sorghum Bread         | Sorghum Bread            | Sorghum Bread     | Sorghum Bread           | Sorghum Bread     |
| CW - 16 % - 24 h | 121.65 (0.21) bcd, A | 53.35 (0.64) bcd, B  | 1535.00 (63.64) e, A    | 1018.50 (28.99) c, B  | 0.37 (0.03) a, B         | 0.45 (0.02) ab, A | 1.15 (0.05) a, B        | 2.09 (0.07) cd, A |
| CW - 18 % - 24 h | 124.73 (3.72) abc, A | 53.75 (1.20) bcd, B  | 2051.00 (93.34) a, A    | 1050.50 (9.19) c, B   | 0.35 (0.02) a, B         | 0.44 (0.02) ab, A | 0.95 (0.08) b, B        | 2.40 (0.21) bc, A |
| HW - 16 % - 12 h | 124.73 (3.72) abc, A | 53.45 (0.64) bcd, B  | 1778.00 (60.81) bcd, A  | 1193.67 (49.14) bc, B | 0.36 (0.02) a, B         | 0.44 (0.01) ab, A | 1.02 (0.07) ab, B       | 2.11 (0.03) cd, A |
| HW - 16 % - 18 h | 114.43 (2.21) de, A  | 56.20 (1.22) abcd, B | 1915.33 (32.62) ab, A   | 1135.00 (38.18) bc, B | 0.34 (0.00) a, B         | 0.45 (0.01) ab, A | 1.00 (0.03) ab, B       | 2.45 (0.00) bc, A |
| HW - 16 % - 24 h | 114.50 (4.95) de, A  | 50.30 (1.73) d, B    | 1647.50 (41.72) cde, A  | 1368.00 (60.67) a, B  | 0.35 (0.02) a, B         | 0.41 (0.01) b, A  | 1.04 (0.08) ab, B       | 1.83 (0.08) d, A  |
| HW - 18 % - 12 h | 114.63 (2.22) de, A  | 51.80 (1.70) cd, B   | 1994.00 (63.64) a, A    | 1196.00 (62.23) bc, B | 0.35 (0.01) a, B         | 0.43 (0.03) ab, A | 1.03 (0.06) ab, B       | 1.82 (0.31) d, A  |
| HW - 18 % - 18 h | 109.50 (1.41) e, A   | 54.77 (2.71) bcd, B  | 1863.50 (84.15) abc, A  | 1233.33 (67.86) ab, B | 0.34 (0.01) a, B         | 0.43 (0.02) ab, A | 0.99 (0.02) ab, B       | 2.06 (0.13) cd, A |
| HW - 18 % - 24 h | 117.63 (1.76) cde, A | 55.27 (1.29) bcd, B  | 1739.00 (73.54) bcde, A | 1045.00 (21.21) c, B  | 0.36 (0.01) a, B         | 0.47 (0.01) a, A  | 1.10 (0.06) ab, B       | 3.25 (0.20) a, A  |
| S - 5s           | 128.45 (0.64) ab, A  | 57.70 (3.12) abc, B  | 1671.50 (21.92) cde, A  | 1156.67 (88.91) bc, B | 0.37 (0.01) a, B         | 0.45 (0.02) ab, A | 1.07 (0.04) ab, B       | 2.01 (0.16) cd, A |
| S - 10 s         | 127.27 (2.78) ab, A  | 59.60 (2.69) ab, B   | 1770.00 (48.08) bcd, A  | 1025.00 (55.76) c, B  | 0.37 (0.02) a, B         | 0.47 (0.01) a, A  | 1.05 (0.03) ab, B       | 2.79 (0.04) b, A  |
| S - 15 s         | 131.60 (2.40) a, A   | 61.77 (1.43) a, B    | 1612.50 (62.93) de, A   | 1017.00 (11.31) c, B  | 0.38 (0.00) a, B         | 0.45 (0.03) ab, A | 1.12 (0.02) ab, B       | 2.47 (0.18) bc, A |

<sup>[b]</sup> CW = cold water, HW = hot water, S = steam. Values in the parentheses are standard deviations. The same lower-case letter in the same column for the specific type of sorghum indicate no significant difference ( $p \leq 0.05$ ). The same upper-case letter in the same row for a given treatment and density type indicate no significant difference.

### **5.3.6. Crumb Texture Profile Analysis**

#### **5.3.6.1. Firmness**

The firmness of white sorghum bread decreased with the tempering condition changing from cold water (1359.83g) to hot water (1229.30g) to steam (1015.92g). Cold water tempered waxy white sorghum also produced bread with greater firmness (465.38g) when compared to bread from waxy white sorghum tempered with hot water (431.38g) and steam (301.96g). Firmness of crumb has been reported to increase with increasing amount of protein in the flour (Storck et al., 2013). Flour from steam tempered white and waxy white sorghum had the least amount of protein when compared to the flour produced from other tempering treatments (Table. 4-4). This could be attributed to the low firmness of sorghum bread produced from steam tempered white and waxy white sorghum. A strong positive correlation was determined between the protein content of the flour and firmness of the bread ( $r = 0.79$ ). No definite trend was observed in the firmness of white sorghum bread with increasing tempering moisture content when grain was treated with cold water or hot water (Table. 5-3). Hot water and steam tempering time also did not affect the firmness of white sorghum bread. The firmness of waxy white sorghum bread increased with increasing steam tempering time. Waxy white sorghum treated with hot water for 24 hours displayed its bread to have greater strength to withstand compression, regardless of the tempering moisture content when compared to other hot water tempering times. White sorghum bread required greater force to compress for the same distance than waxy white sorghum bread when comparing every individual tempering treatment. This could be attributed to lower bran contamination in white sorghum flour.

It is preferred gluten-free bread to have high firmness. White sorghum bread produced cold water tempered white sorghum to 18% m.c. (w.b.) for 24 hours and white sorghum tempered with

hot water for 18 hours to bring to final m.c. 16% (w.b.) displayed high firmness 1398.79g and 1293.46g respectively. Similarly, waxy white sorghum tempered with cold water for 24 hours to bring to final m.c. 18% (w.b.) produced bread with high firmness value (611.28).

#### **5.3.6.2. Resilience**

No relationship has been observed between resilience and type of tempering, tempering time or tempering moisture content for both white and waxy white sorghum bread (Table. 5-3). However, for every individual tempering treatment, white sorghum bread showed greater capacity to come back to original shape after compression than waxy white sorghum bread. This could also be related greater protein content in waxy white sorghum flour ( $r=0.34$ ).

For white sorghum bread, the resilience increased with increasing tempering moisture content when treated with cold water. The different applied tempering conditions also played no role in the resilience of white sorghum bread. Similarly, waxy white sorghum bread produced from cold water tempered (18% m.c., w.b.) sorghum displayed greater resilience when compared to the bread produced from cold water tempered for 24 hours to bring to final m.c. 16% (w.b.). Cold water, hot water or steam tempering did not play any significant effect on the resilience of waxy white sorghum bread. Both white and waxy white sorghum tempered with hot water for 18 hours to bring to final m.c. 16% (w.b.) also had high resilience.



Table 5- 3. Texture profile analysis of white and waxy white sorghum bread<sup>[c]</sup>

| Treatment        | Firmness (g)          |                      | Resilience (%)      |                     |
|------------------|-----------------------|----------------------|---------------------|---------------------|
|                  | White Sorghum         | Waxy White Sorghum   | White Sorghum       | Waxy White Sorghum  |
| CW - 16 % - 24 h | 1234.81 (9.81) ab, A  | 319.49 (27.86) de, B | 47.63 (0.60) abc, A | 34.75 (0.10) bcd, B |
| CW - 18 % - 24 h | 1398.79 (23.13) a, A  | 611.28 (20.95) a, B  | 54.25 (0.20) a, A   | 36.69 (0.17) ab, B  |
| HW - 16 % - 12h  | 1241.46 (15.44) ab, A | 514.51 (7.05) b, B   | 51.07 (1.17) abc, A | 38.36 (0.33) a, B   |
| HW - 16 % - 18 h | 1293.46 (35.43) ab, A | 375.10 (22.26) cd, B | 50.22 (0.36) abc, A | 32.44 (1.34) ed, B  |
| HW - 16 % - 24 h | 1283.26 (91.97) ab, A | 584.15 (15.25) a, B  | 50.58 (1.24) abc, A | 35.14 (0.25) bc, B  |
| HW - 18 % - 12 h | 1234.53 (98.34) ab, A | 324.52 (20.54) de, B | 42.42 (0.55) c, A   | 35.22 (1.03) bc, B  |
| HW - 18 % - 18 h | 1093.43 (12.42) bc, A | 312.15 (20.01) de, B | 46.71 (0.31) abc, A | 35.81 (0.23) bc, B  |
| HW - 18 % - 24 h | 1229.65 (49.86) ab, A | 505.98 (18.27) b, B  | 52.04 (2.04) ab, A  | 33.75 (1.22) cde, B |
| S - 5s           | 1089.31 (18.53) bc, A | 220.86 (18.40) f, B  | 46.47 (5.00) abc, A | 34.43 (0.14) bcd, B |
| S - 10 s         | 1026.78 (12.42) bc, A | 283.75 (15.61) e, B  | 43.71 (3.28) bc, A  | 31.67 (0.85) e, B   |
| S - 15 s         | 1003.12 (107.48) c, A | 410.38 (14.14) c, B  | 50.31 (0.74) abc, A | 34.30 (0.34) bcd, B |

<sup>[c]</sup> CW = cold water, HW = hot water, S = steam. Values in the parentheses are standard deviations. The same lower-case letter in the same column for the specific type of sorghum indicate no significant difference ( $p \leq 0.05$ ). The same upper-case letter in the same row for a given treatment and density type indicate no significant difference.

#### 5.4. Conclusion

Smaller diameter crumb cells with thinner walls and greater number of crumb cells are preferred in a good quality white pan bread. Similar attributes are preferred in GF breads too as smaller cells in larger number produce loaves with greater specific volume. Though a specific trend was not observed with increasing tempering moisture content or tempering time period, the type of tempering produced a significant effect on the bread characteristics. Steam tempered sorghum produced bread with higher cell brightness which is attributed lowest bran and ash content. The undesirable bread characteristics of waxy white sorghum bread could be majorly attributed to the presence of greater starch damage in the flour. The presence of greater amount of damaged starch content has proven to improve the water absorption capacity of the flour from previous research. Thus, further study on the dough characteristics of the flour produced from these tempering conditions would help to study the benefits of presence of greater starch damage in the flour. Other factors like greater ash content and greater protein content in waxy white sorghum flour could have possibly increased the water absorption capacity of the flour resulting in poor bread characteristics from waxy white sorghum flour. Thus, the use of expensive ingredients like hydrocolloids for improving the viscosity of the batter by trapping more air may not be a suitable addition to make waxy white sorghum bread. A revision of the ingredients used to make waxy white sorghum bread with better bread properties could be an important consideration for the future study. Cold water tempered white and waxy white sorghum to final m.c. 18% (w.b) and, white and waxy white sorghum tempered with hot water for 18 hours to bring to final m.c. 16% (w.b.) produced bread with greater number crumb cells and high firmness and resilience. White sorghum bread produced the most desirable bread characteristics with good volume index, greater number of crumb cells, thinner cell walls, crumb cells with smaller diameter, high firmness and

resilience. This is attributed to lower bran contamination/ash content and lower starch damage of white sorghum flour produced from all tempering treatments when compared to waxy white sorghum flour.

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## **Chapter 6 – SUMMARY OF CONCLUSION AND DISCUSSION**

### **6.1. Restatement of Thesis Goals**

Celiac disease and other gluten-related disorders have been gaining popularity with years. Several people are diagnosed with these disorders which has greatly impacted the global gluten-free market. Thus, there is a continuous need for varying gluten-free products. Sorghum being gluten-free can be suitable substitute for gluten containing products. Besides being gluten-free, it is also nutritious and can be easily cultivated. United States is also the leading producer of sorghum in the world. However, most of it is used in the production of animal feed or ethanol making.

The unique structure of sorghum has been a challenge for milling. With several milling techniques applied in the past to procure sorghum flour, roller milling has proven to be more advantageous than the conventional milling methods. The milling techniques like hand pounding, Abrasive Decortication Hammer Milling (ADHM) and pin milling produced flour with greater starch damage, discoloration and greater bran contamination. Inconsistency in flour extraction rate and quality limits the commercialization of gluten free sorghum products. Though roller milling was proven to be more efficient in terms of separating bran and endosperm, there have been issues with bran contamination in flour. In order to improve the quality of flour produced from roller milling, proper tempering techniques are imperative as a pre-milling process. It helps to toughen the pericarp and mellow the endosperm and facilitates easier separation of bran. Besides this using the advantage of roller milling in terms of flour quality, thus developing a roller milling flow sheet for sorghum saves on capital investments.



This study mainly focusses on studying the effects of tempering on white and waxy white sorghum kernel, milling, flour, and bread properties. As stated in Chapter 1, the objectives of this research are:

2. To determine the effect of tempering methods on white and waxy white sorghum kernel properties.
3. To develop novel mill flow sheet using laboratory-scale roller mill for milling white and waxy white sorghum
4. To determine the effect of tempering methods on white and waxy white sorghum flour and bread making properties.

## **6.2. Project Overview**

The thesis begins with Chapter 1 Introduction, where the foundation of this research is explained as the problem statement. The research is justified by the prevalence of gluten-related disorders, advantages of sorghum, benefits of using roller milling for sorghum and the importance of tempering as a pre-conditioning technique to roller milling. The research hypothesis is tempering affects moisture penetration into the kernel and the milling behavior which in turn affects properties of flour and baking characteristics.

The second chapter consists of the Literature Review. This chapter reviews the peer reviewed publications on celiac disease and other gluten-related disorders, gluten-free market, structural characteristics and chemical composition of sorghum. Additionally, studies on the importance of sorghum and its processing: tempering, milling, baking, are included.

Chapter 3 elaborates the research on first objective. This chapter explains the 11 tempering conditions used on white and waxy white sorghum: cold water (to final m.c. 16% and 18% (w.b.) each for 24 hours), hot water (to final m.c. 16% and 18% (w.b.) each for 12, 18 and 24 hours) and steam tempering (at 20 psi for 5, 10 and 15s). The effect of these tempering conditions on physical properties (bulk, tapped and true density), mechanical properties (SKCS characteristics and abrasive hardness) and frictional properties (coefficient of static and rolling friction; and angle of repose) of the kernels are studied.

Chapter 4 focuses on the second objective i.e., developing a roller milling flow sheet using the laboratory-scale roller mill. Besides the development of the milling flowsheet, the effects of the tempering conditions on the milling behavior of white and waxy white sorghum are also studied. The effects of these tempering conditions on the milling outcomes (flour, bran, shorts and reddog) are determined. Impact of these tempering conditions on the proximate composition (total starch content, protein content, ash content, crude fat content, crude fiber content, and damaged starch content) and physical properties (color, bulk, tapped and true density) of the procured flour are also evaluated.

Chapter 5 focuses on studying the effects of the tempering conditions on the baking characteristics of white and waxy white sorghum flour. Bake loss, volume index, internal crumb structure (slice brightness, average cell number, cell wall thickness and cell diameter) and, crumb texture profile analysis (firmness and resilience) were studied.

### **6.3 Discussion of Major Findings from this Research**

The tempering conditions significantly affected the kernel properties. The kernel properties, milling characteristics and baking quality of the sorghum kernels were not significantly

affected by tempering time using hot water. This is attributed to the ineffectiveness of indirect heat treatment provided during hot water tempering. Tempering time had an evident impact on the mentioned properties in case of steam tempering. Heat and pressure during steam tempering caused structural changes in the pericarp, causing it to easily scrape off from the kernel during milling. Steam tempering of white and waxy white sorghum at 20 psi for 15s helped to efficiently remove maximum bran and provide flour with the least ash content. However, the protein content and flour yield from this tempering condition were heavily compromised. The moisture content of the kernels and flour following steam tempering is a major concern. Moreover, the feasibility of steam tempering at an industrial scale is limited due to greater investments in terms of maintenance and energy.

The developed flowsheet produced flour in which 98% of the total flour was less than 212 $\mu$ m which satisfies the requirement by CFR to be claimed as flour. The developed flow sheet produced an average of 60.24% flour from white sorghum and an average of 39.8% of flour yield from waxy white sorghum. The higher production of shorts on milling waxy white sorghum is attributed to the internal structural composition of waxy white sorghum. Considering the flour yield, bran extraction and proximate composition, the following two are the best tempering treatments on white and waxy white sorghum: Cold water tempering for 24 hours to bring to final m.c. 18% (w.b.) and hot water tempering for 18 hours to bring to final m.c. 16% (w.b.).

Bread properties did not particularly show a definite trend with increasing tempering moisture content or increasing tempering time period, the type of tempering (cold water, hot water and steam) provided significant effect on the bread properties. The presence of excess ash content and bran contamination in waxy white sorghum flour produced from all tempering conditions, has caused it to produce bread with undesirable characteristics. White sorghum flour produced from

white sorghum tempered with all 11 tempering treatments produced bread with greater volume index, greater number of crumb cells, thinner cell walls and smaller sized cells.

The overall conclusion from the entire study is: considering better flour yield, proximate composition in terms of high protein and low ash content, low damaged starch content, bread with high volume index, greater resilience, greater slice brightness and, greater average number of crumb cells; the two best tempering treatments for white and waxy white sorghum are tempering with cold water for 24 hours to bring to final m.c. 18% (w.b.) and, tempering with hot water for 18 hours to bring to final m.c. 16% (w.b.).

#### **6.4. Future Work**

- The wax on the surface of the kernel could also play a significant role in the water absorption during tempering. Hence, it is essential to study the characteristics of the surface wax and apply proper abrasion techniques to remove it to better understand the movement of water inside the kernel during tempering.
- A detailed research on the impact of tempering on the textural and breakage characteristics of white and waxy white sorghum kernel can be advantageous.
- Compositional analysis of other milling fractions could possibly help in identifying co-products from the sorghum milling industry.
- Improvising the tempering conditions on waxy white sorghum kernel to improve the milling yield is recommended.
- Performing rheological studies on dough could help in prediction of baking properties of the sorghum flour.

- Extending the studies to pilot-scale milling throw light on the feasibility of developed flow sheet for commercialization.
- Studying the possibilities of developing other gluten free products with sorghum flour.