OPTIMUM STEEPING PARAMETERS OF CORN GRITS

by

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B.S., University of Peradeniya, 1981

A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

AGRICULTURAL MECHANIZATION

KANSAS STATE UNIVERSITY
Manhattan, Kansas

1988

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

ACKNOWLEDGMENTS

I would like to express my sincere appreciation and thanks to my former major Prof. Dr. S. R. Eckhoff and major Prof. Dr. C. K. Spillman for their invaluable help, support, guidance and encouragement throughout the study. Gratitude is extended to Dr. D. S. Chung, and J. W. Slocombe for serving as my committee members.

I wish to thank Dr. J.M. Faubion for his great help and support, especially when analyzing samples. Special thanks and appreciation are due to Dr. R.C Hoseney and Dr. P. A. Seib for contributing their valuable time to the discussion of various aspects of the study.

I would like to express my appreciation to Dr. W. D. Eustace for his help during the dry milling process. Thanks are also due to Marty Criswel, who worked with me throughout the study, for his support and suggestions during the study. I would like to thank Mark Inglen for his help during quality test of samples and Indrasiri Fernando for his help during statistical analysis.

The author's M.S. study has been sponsored by the Rotary Foundation of Rotary International in USA. This support is greatly appreciated.

INTRODUCTION

Starch, an abundant renewable raw material, is used in the production of a wide variety of industrial and food products. More than 14 million tons of starch is produced annually by the corn wet milling industry and processed into corn syrup, chemicals, plastics, papers, drugs, brewing and confectionery products. The corn starch is produced by a wet milling process in which whole corn kernels are steeped in sulfurous acid for about 24 hours to facilitate the separation of different components.

The most important and expensive single step in the corn wet milling process is steeping. The yield and quality of starch and thus its products depend on the conditions of the steeping process. The yield and purity of starch is directly related to the degree of disintegration of the endosperm protein. Therefore, the path by which sulfurous acid enters the kernel, and the permeability of the pericarp, tip cap, seed coat and aleurone layer are important factors of steeping. It is, however, well known that water moves rapidly through the spongy layers of the pericarp to the top of the kernel before diffusing across the seed coat and aleurone layer to the endosperm and germ. This indicates that the rate of water and sulfurous acid absorption by the germ and endosperm is controlled by the

resistance of the seed coat and aleurone layer.

Several investigators have used different chemicals, various steeping temperatures, high pressure disintegration techniques, etc. attempting to reduce the steeping time by increasing the rate of water penetration into the corn endosperm. The slow diffusion rate of water could be increased by placing corn grits, instead of whole corn kernels, in direct contact with steeping water. In this way, sulfurous acid might disperse more completely and rapidly the protein matrix holding starch granules thus reducing the time and cost of steeping.

OBJECTIVES

1). To determine the appropriate sulfur dioxide level and steeping time required to steep corn grits in a dynamic batch steep.

2). To determine the extent of starch damage created by degermination and/or steeping in the wet milling process of corn grits.

LITERATURE REVIEW

Starch processing is one of the oldest industries and its history goes back about 3000 years. Starch was obtained from other grains and root vegetables, like potatoes, long before the first corn refiner milled his first batch of corn. In the first few decades of the 19th Century, wheat and potato starch were made in homes and small mills. The first U.S. starch factory was built at Hillsborough, New Hampshire, in 1802. It manufactured potato starch for use in cotton mills. In 1840, an Englishman, Orlando Jones, patented a process of using alkali to separate starch. In 1841 he obtained U.S. Patent No. 2000, for the corn refining industry. The process covered extraction of starch from any appropriate plant source. It was soon applied to corn because corn was, and still is, the best available source of starch. In 1842, Thomas Kingsford, who was the first to extract starch from corn on a commercial basis, founded the corn refining industry (Inglett 1970).

Dry milling of corn is primarily concerned with separation of the anatomical parts of the grain. Wet milling strives for the same separation but also goes a step further and separates some of those parts into their chemical constituents. Thus, the primary products of corn wet milling are starch, protein, oil and fiber instead of

bran, germ, and grits in the dry milling industry (Hoseney 1986).

FACTORS AFFECTING WET MILLING QUALITIES OF CORN

Method of Drying

Brown et al. (1981) studied the suitability of corn exposed to high drying temperature at different moisture contents for wet milling. They found that the yield and quality of starch produced by wet milling of corn is often low when the corn has previously been subjected to high temperature during artificial drying. Bras (1982) studied the relationship between drying conditions and starch yield. He found that beyond 90 ^{o}C the starch yield decreases as the temperature of the drying air increases. At 140 ^{o}C the decrease in starch yield was more than 11%. This correlated with the results determine by Cox et al. (1944). Their data shows that when corn was artificially dried at 180 to 200 ^{o}F (82 - 93 ^{o}C) it was impossible to separate all of the gluten from the starch.

This was similar to the conclusions made by Kent (1983) in which he mentioned that the temperature of the drying air should not exceed 130 ^{o}F (54 ^{o}C) in order to obtain corn with better wet milling properties. He also found that at

higher temperatures some changes occur in the protein whereby it swells less during steeping and tends to hold starch more tenaciously than in grain not artificially dried or dried at low temperatures. In addition, if dried at a temperature above 130 ^{o}F the germ becomes rubbery and tends to sink in the ground corn slurry resulting in a higher oil content in the starch. The Findings of Watson and Sanders (1961) confirm the above conclusions. They found that corn dried from 32 - 12% moisture in air at 120 ^{o}F (49 ^{o}C) gave normal starch release, but corn dried at 200 ^{o}F (93 ^{o}C) gave very little starch release under any steeping condition. Corn dried at 180 ^{o}F (82 ^{o}C) and steeped at 52 ^{o}C with 0.2% SO_2 gave two-third as much as starch as the corn dried at 120 ^{o}F (49 ^{o}C).

Similar results have been observed by Freeman (1973). He found that the method of drying has the greatest effect on millability of corn in addition to inherent grain characteristics. High temperature used in artificial drying of shelled corn was extremely detrimental to the corn endosperm. The protein appeared to be "case-hardened" and was not as easily disrupted by steeping in SO_2 solutions. Consequently, starch yield was reduced and protein content of isolated starch was decreased. Corn dried from 30 to 15% moisture in one pass through a high temperature dryer caused a 25% reduction in production capacity of a modern wet

milling plant, poor dewatering of coarse fiber, higher level of starch in gluten with a correspondingly lower starch yield, higher protein content of isolated starch and lower viscosity of starch.

Amylose Content

The most important study on the relationship between amylose content and wet milling qualities of corn was done by Anderson et al. (1960). They studied the wet milling qualities of corn containing 49 and 57% amylose starch. They observed that a 64.4% yield of starch containing 0.51% protein could be recovered from ordinary corn, as compared with a 43.5% yield of starch containing 0.7% protein from high amylose corn . An unusually large swelling of the corn kernels was observed during the steeping of high amylose corn. These kernels exibited an increase of 128% of their original dry volume, as compared with a 63% increase in volume during the steeping of ordinary corn.

Inglett (1970) has made the same conclusions regarding corn with high amylose content. He found that the starch yield, based on original grain weight, was lower for the amylomaize samples than that of ordinary corn. This difference would be expected since amylomaize contains a lower amount of starch. Starch recovery, based on total

starch content, ranges from 70 to 83% for the amylomaize hybrids as compared to 87% for ordinary dent corn. The lower starch recovery from amylomaize could be due to difficulties in separation of small and irregularly shaped starch granules present in these varieties.

Lysine Content

An extensive study on the wet milling properties of high lysine corn was done by Watson and Yhal (1967). Their data showed that high lysine corn swelled to as much as twice the volume of the normal corn during steeping and contained 52.3% moisture as compared with the 42.8 to 44.6% moisture for normal corn. Wet milling of opaque-2 corn produced high yields of solids in steep water, germ and fiber and low yields of gluten and starch. Starch yield of opaque-2 corn was 64.4% while that of normal corn samples were 71.4 to 73.6%, respectively . However, the starch recovered from the opaque-2 corn was similar in all properties to the starch recovered from normal corn.

Grain Preservatives

Preliminary laboratory wet milling studies with corn preserved with propionic acid and a mixture of propionic and acetic acids have indicated some potential disadvantages to

the wet miller. After several months of storage at room temperature, kernels began to darken and a large proportion of yellow endosperm pigments were destroyed by oxidation. However, grains treated with the acids were very soft and milled easily. Separation of starch and protein were exceptionally good (Freeman 1973).

STEEPING

The soaking of corn in sulfurous acid under specific conditions of temperature and time period in order to get optimum milling separation of components is called steeping. The softening action that results from steeping is important since it determines the efficiency of the milling operation with respect to the recovery of components. It is also complex because of the difference in chemical composition of the various grain constituents or parts which are to be conditioned. The moisture content of the corn increases rapidly at first to about 35-40% and then levels off at about 43-45% during steeping. At this moisture content the hull and germ are readily removed by a coarse grinding operation. However, even at a satisfactory moisture level the starch cannot necessarily be separated from the gluten in which it is embedded and it is apparent that either (a) some change in the physical nature of the protein is

required or (b) the dissolution of the cementing substance holding granules in the protein is required. Existing information proves that the protein itself is the cementing agent (Kerr 1950).

According to Cox et al. (1944) and Kerr (1950) several objectives are to be achieved in the steeping process. The most important of which are as follows :

1). To soften the corn kernel so the various components of the grain such as hull (fiber), the germ (which contains nearly all of the oil), the gluten and the starch may be separated from each other with a higher degree of efficiency.

2). To reduce or inhibit the activities of micro-organisms during steeping.

3). To remove solubles, mainly from the germ.

4). To react bisulfite with disulfide bonds in the matrix proteins of the corn and reduce the molecular weight of the protein, making them more hydrophilic and thus more soluble.

Penetration of Corn Kernel by Steep Water

One of the most extensive studies on water penetration into the corn kernel during steeping was done by Cox et al. (1944). They found that the steeping liquid enters into the

kernel at the basal end and travels rapidly upward just under the seed coat. Water apparently reaches the top of the kernel before it starts to penetrate through the aleurone layer to the starch granules of the endosperm to any appreciable extent.

Fan et al. (1963) derived a theoretical diffusion equation, based on Fick's Law, to represent the rate of water absorption into a corn kernel. They have used arbitrary shapes of grain for their calculations. They found that the derived equation was well correlated with the experimental data for wheat. They mentioned that the diffusivities of water in corn and sorghum are of the same order of magnitude as those in wheat.

Wagoner (1948) studied the changes occurring in corn with steeping time. He observed that the corn was very hard for up to 12 hours of steeping. The protein matrix was intact, the starch was held tenaciously, and the cellulose walls were very firm. Only slight changes were noticed until the corn had been steeped for about 24 hours. At this time the sulfurous acid content of the steep water began to decrease quite rapidly and the protein matrix in the floury endosperm began to break up, freeing starch granules. At 36 hours the protein network was dispersed throughout the horny endosperm. Continued steeping enhanced this disorganization

until the protein network had disappeared even around the small granules next to the aleurone layer. It was noticed that there was no protein matrix, but the cellulose cell walls were intact when corn was steeped for 50 hours. Most of the protein network had disappeared in the sulfurous acid and the remainder had folded back around the cellulose walls. The starch was easily separated from the protein giving starch of low nitrogen content. At all temperatures, absorption rate was slower in sulfur dioxide solution than in water in the initial period of steeping, but this was reversed as absorption time was increased. The higher the steeping temperature the sooner the reversal took place.

Watson et al. (1951) mentioned that the embryo of the corn kernel was saturated with water after four hours of steeping while endosperm was saturated after eight hours. The maximum moisture content of 42 - 48% was attained after 8 - 12 hours. At this point, however, the kernel was not softened. Wagoner (1948) also studied the relationship between steeping time and moisture content of corn. Corn reached its maximum moisture content of about 45% after about 45 hours of steeping. Wahl (1970) concluded that corn grain absorbed a substantial amount of sulfur dioxide during the first phase of steeping regardless of the initial concentration. Maximum absorption occured after 10 hours.

An excellent work to modify the existing method of corn steeping was done by Meuser et al. (1984). They used a high pressure disintegration method to decrease the steeping time. Corn was steeped at 50 ^{o}C and 218 psia for 2 hours. They found that the moisture content of corn reached 40% and the starch yield was 62.5%. They also found that use of SO_2 was not necessary in the modified process. However, Wahl and Franzke (1969) concluded that the chemical reactions which occur during the steeping process were mainly due to sulfur dioxide, and further, those connected with the production of lactic acid as a result of microbiological processes. These alteration and degradation reactions were important in the wet milling process. The starch granules are embedded in a protein matrix in the endosperm of the corn kernel. An extensive loosening and separation of this matrix is the pre-requisite for an effective separation of starch and protein by subsequent processing. The method used by Meuser et al. (1984), therefore, would have been more appropriate if they had planned to take advantage of SO_2 in steeping.

In their study, Hassanean and Wahed (1986) used a new method to shorten the steeping time. They found that steeping of corn by circulating the corn with the steeping liquor was more efficient in removing the water soluble components, and decreasing the steeping time compared to the

usual counter-current steeping method. They also found that the starch yield could be increased by about 1% compared to the usual method. The protein content of the starch could be decreased to 0.3%.

Chemical Treatments in Steeping

Kerr (1950) used different types of chemicals, namely, acetic acid, lactic acid, hydrochloric acid, sodium benzonate, halogen compounds (such as monochlorobenzene and o-chlorotoluene), formaldehyde, etc., to determine their suitability in the corn steeping process. He found that Lactic acid tends to soften the gluten while other chemicals have no significant effect in the steeping process. However, the presence of large amounts of lactic acid in the steeping liquor is undesirable due to several reasons. It promotes the dissolution of protein to an undesirable extent and the lactate ion has an adverse effect on the sedimentation of starch from finely ground mixtures of starch and gluten.

Cox et al. (1944) also studied the action of lactic acid as a steeping agent. This was important since a considerable amount of this acid is formed by microbial action and it is believed by some industrialists that lactic acid promotes some favorable reactions in the industrial

steeping process. They found that the extend of protein disintegration was equal to that of a water steep and softening action roughly equal to that of a 0.1% sulfur dioxide steep. Protein disintegration of both 0.2% and 1% lactic acid steeps were equal to that of a 0.1% sulfur dioxide steep and softening was equal to a 0.2% sulfur dioxide steep. Protein disintegration during a steep with a mixture of 0.1% sulfur dioxide and 0.1% lactic acid was equal to that of 0.2% sulfur dioxide steep but the softening action was slightly higher than the latter. They did conclude that lactic acid has relatively little ability to disintegrate the protein matrix but does exert an appreciable influence in softening the corn kernel.

A similar study was done by Roushdi et al. (1981) to investigate the role of lactic acid in corn steeping and its relation to starch isolation. They found that the solubilization and diffusion of protein was stronger in corn grains steeped completely in old sulfur dioxide or fresh sulfur dioxide with lactic acid. Such treatments gave the highest starch yields with lowest protein contents in the isolated starch. The low amount of starch content in hull and fiber confirmed the important role of lactic acid in addition to the sulfur dioxide.

However, at present, sulfur dioxide is almost

universally employed due to its low cost, pronounced germicidal properties and other reactions which are favorable in steeping. The use acetic acid, lactic acid or HCl do not give comparable results. The addition of lactic acid to sulfur dioxide, however, increases the softening action.

Sulfur Dioxide Treatment

The most extensive study on the function of sulfur dioxide as a steeping agent has been been done by Cox et al. (1944). These investigations showed that starch was embedded in and tightly held by a protein network and the network swells and tends to form tiny globules of hydrated protein during steeping. Protein tended to disperse and finally the dispersed protein became so weak that it collapsed against the cell walls and showed little evidence of the original network after removal of the starch granules. Sulfur dioxide greatly accelerated this process.

Cox et al. (1944) also indicated that both high sulfur dioxide concentration and high steeping temperature lead to increased protein disintegration and dispersion. The relationship between protein dispersion and starch recovery is probably due to the fact that the more dispersed the proteins the less starch it can carry into the tailing or

gluten fraction, and, hence, the greater the amount of prime-quality starch. The results on the disintegration of protein by sulfur dioxide were corroborated by the behavior of the gluten fraction in corn from an overnight steep at 49 ^{o}C in 0.2% sulfurous acid and in distilled water. In both instances the protein was still in microscopic aggregates, but from the sulfur dioxide steep they were only about one fourth the diameter of those remaining after the water steep.

In their study, Cox et al. (1944) used acetic and hydrochloric acids, in concentrations equivalent to a 0.2% sulfur dioxide solution, to determine whether the softening action of sulfurous acid was due to its acidity. They observed that corn steeped in an acetic acid solution was somewhat hard to grind and separation of starch from gluten was difficult. After the hydrochloric acid steep the corn could be ground with difficulty. Separation of starch from gluten was fairly easy, but only a 56% recovery of starch, containing 0.07% nitrogen (d.b.), was obtained. Similar results have been obtained when acetic and hydrochloric acid steeps at pH 1.68 to 1.70, equivalent to that of 0.2% sulfur dioxide, were used. A corn sample steeped for 4 hr in sulfur dioxide solution was as easy to process as that steeped for 24 hr in either acetic or hydrochloric acid

solutions. They concluded that acidity of sulfur dioxide plays no more than a minor role in softening the corn kernel and loosening gluten from starch granules.

Fan et al. (1965) compared the rates of absorption of water by corn kernels with and without dissolved sulfur dioxide. They found that adding sulfur dioxide to water has two extreme effects on absorption rate, a retarding effect at the onset of steeping and then an acceleration effect. At all temperatures (10, 25, 38, 49, 60 and 71 ^{o}C), absorption rates were slower in sulfur dioxide solution than in water in the initial period of steeping, but the rates were reversed as absorption time increased. The higher the steeping temperature, the sooner the reversion took place. The acceleration in absorption rate increased with increasing temperature up to the gelatinization temperature of of the starch.

Krochta et al. (1980) studied the effect of sulfur dioxide concentration and water to corn ratio in the corn steeping process. They found that the highest starch yield of 70.3% could be obtained with a sulfur dioxide concentration of 0.2% and a water-corn ratio of 2 : 1. Watson et al. (1955) investigated the changes in sulfur dioxide concentration, dry substances, reducing sugar content, relative bacterial activity (RBA) and PH level with

time during the steeping process. The sulfur dioxide concentration had little effect on change in solids content in the steep water. Sulfur dioxide had a marked effect on relative bacterial activity, reducing sugar concentration and lactic acid concentration of the steep water. The total nitrogen and amino nitrogen contents increase with time in all sulfur dioxide concentrations. However, a higher concentration of sulfur dioxide decreases the total nitrogen and amino nitrogen contents in the steep medium.

The effect of temperature in the steeping process was studied by Cox et al. (1944). They used several 0.2% sulfur dioxide steeps at temperatures below (38, 43) and above (52, 55) 49 ^{o}C. Differences in starch recovery were not significant under laboratory processing conditions although processing was easier the higher the temperature used. It is well known by the industry, however, that the temperature of the steep employed at the factory has a profound effect upon both the ease of processing and the amount of starch recovered.

Methods of Sulfur Dioxide Treatments

According to Kerr (1950) there are two methods of sulfur dioxide treatment.

1). Wooden vat : The most elementary method of steeping

consists of partly filling a wooden vat holding about 2000 to 2500 bushels of corn and covering the grain with warm water containing sulfur dioxide. The temperature is maintained at about 50 ^{o}C for 40 to 48 hours and after which the steep water is drained from the corn.

2). Counter current flow : About 10 wooden vats arranged in series are used in this method. Steeping is done in a series of wooden or stainless steel tanks in which lactic acid fermentation is controlled by a counter current flow of water mixed with sulfur dioxide. The acidic steep water containing 0.1 to 0.2% sulfur dioxide, is heated to about 50 ^{o}C, and put on the corn which has been longest in the steeping cycle. The acid water is transferred so the oldest corn is steeped with water containing the highest amount of acid and newest corn with water containing the lowest amount of acid.

Effect of Sulfur dioxide on quality of starch

Cox et al. (1944) found that the concentration of sulfur dioxide in steeping medium affects the viscosity of corn starch. Nitrogen (or protein) and phosphorus contents of starch increase as sulfur dioxide concentration of the

steep increase. The amount of methanol extractable fat, however, was found to be relatively constant. Starch extracted from corn after steeping in 0.1% sulfur dioxide was significantly lower in viscosity than that extracted after steeping in water. The higher the sulfur dioxide concentration of the steep, from 0.1 to 0.4% , the lower the viscosity of the starch extracted. Viscosity tended to vary inversely with the nitrogen and phosphorus contents of the starch. Sulfur dioxide concentration of the steep had no significant bleaching effect on starch.

Growth of Micro-Organisms

Kerr (1950) studied the effect of sulfur dioxide in respect to its germicidal properties in wet milling. He found that free sulfur dioxide, or sulfurous acid, was an effective inhibiting agent of micro-organisms even at low concentrations (0.02 - 0.03%). Its efficiency could be markedly increased by raising the temperature over the range from 30 to 50 ^{o}C. However, a considerable portion of sulfur dioxide added to the steep does not remain free in the steeping liquor. It reacts to form bisulfite and other compounds with amino groups and carbohydrates. These combined chemical forms are found to be less effective sterilizing agents than sulfur dioxide.

Wahl and Franzke (1969) summarized the findings of several other researchers on the bacterial activities during steeping. He mentioned that a considerable amount of bacterial activity was possible only when the sulfur dioxide content was below 0.01%. However, the activity depended on both primary factors (maize microflora) and secondary factors (condition of steeping, infection of process water etc.) which adversely influenced the steeping process. The bacterial activity had great influence on the quality of the steep water and hence on the quality of wet milled products. The period of active microbial growth was determined by the amount of amino-nitrogen in the steep water.

Wahl and Franzke (1970) found that a considerable increase in lactic acid concentration occured with the decrease of sulfur dioxide concentration to 0.04 - 0.06% after 6 - 8 hours of steeping. They felt that in that condition the corn kernel was a good substrate for bacteria since the inhibiting action of sulfur dioxide was reduced to a considerable extent. Watson et al. (1955) also mentioned that lactic acid plays an important role in controlling the growth of micro-organism during steeping. Lactic acid was an essential part of the buffer system that keeps the steep water near pH 4. This level of pH inhibits the growth of micro-organisms.

Limitations of Sulfur Dioxide Treatment

The main objections to the use of sulfur dioxide arise from the fact that at the temperature and pH required to secure its beneficial effects, the sulfur dioxide volatilizes from aqueous solutions and that these solutions are acids. The corrosive action of acid is high not only on equipment but also on structural parts of buildings. The acid tends to modify the starch by a hydrolytic action which is first noted by a significant loss in potential paste viscosity of the starch.

SEPARATION OF GERM

After steeping is completed, the steep water is withdrawn. The corn is first passed through a degerming mill. The primary objective of this milling is to crack and open the kernel to free the germ without breaking or crushing the germ (Inglett 1970). The density of the slurry with ground material is adjusted to 1.06 - 1.08 sp. gr. by addition of starch and/or water. The germ will float while the grits and hulls will sink at this specific gravity range. The slurry containing germ, hull and endosperm parts, is passed through liquid cyclones or hydroclones to separate germ from other components. There are some old floatation types of germ separators also in use. The

separated germ is washed to remove starch and gluten (Inglett 1970 and Kerr 1950).

SEPARATION OF FIBER

The milled slurry, containing the ground starch, gluten and hulls is passed through a series of hexagonal reels where the coarse fibers are removed. The starch content of the coarse fibers is less than 7% in modern wet milling plants. Recover of this amount of starch is economically not feasible. The materials going through the reels still contain some fibrous materials, the fine fiber. The fine fibers are removed from the starch and gluten by passing the slurry over gyratory shakers fitted with fine nylon cloth. Separated fibers are rewashed several times to obtain maximum starch recovery (Inglett (1970).

SEPARATION OF STARCH FROM GLUTEN

The specific gravity of the slurry of starch and gluten is adjusted to 1.04 by adding starch or water for better separation of the starch. According to Kerr (1950) several methods can be used to separate starch from gluten.

1). The most elementary method for separating starch from the slurry is to allow the starch to settle for several hours in a vat and then draw off water which contains a

higher amount of gluten.

2). The second method of separation is to introduce the slurry of starch and gluten into a top of a cone shaped vat and to draw off the starch as a concentrated slurry at the bottom.

3). The principles of floation have been developed and applied with considerable success to separate starch in recent years. By the use of several frothing agents and disperssents either gluten or the starch can be floated.

4). Sedimentation of starch from gluten by allowing the slurry to flow continuously along an inclined table is now almost universally employed to make the separation. The dimensions, inclination and flow rate of the slurry on the table are determined by the characteristics of the slurry.

Kerr (1950) pointed out some factors which affect the separation of starch from gluten :

a). Physical state of gluten

b). pH of the slurry

c). Ionic strength of the liquid

d). Temperature of the slurry

e). The rate of flow of the liquid, and

f). The characteristics of the starch table, such as ratio of width to length, smoothness of the surface

and inclination of the table.

Kerr (1950) mentioned the desired physical state of gluten was attained by proper steeping and milling. A satisfactory range of acidity for tabling was a pH between 3.8 and 4.2. Separation became more difficult when the pH value was outside of this range. The starch sedimentation became easier when a low density slurry was used. Within operating limits (75 - 110 ^{o}F), the higher the temperature the greater the efficiency of table separation since an increased in temperature reduced the apparent density of the slurry. When the slurry of starch and gluten was passed over the table at a too high rate some starch passed with the gluten. When the rate was too slow, gluten settled with the starch. The range of rates of flow at which starch was obtained at the highest purity and yield were very narrow.

Wet Milling of Corn Grits

A number of studies have been done by different investigators (Cox et al., 1944; Chu, 1961; Fan et al., 1963; Hassanean and Wahed, 1986; Krochta et al., 1981; Roushdi et al., 1981; Wagoner, 1948; Wahl and Franzke, 1970; Watson, 1961; etc.) to find a better method of steeping in order to reduce steeping time by increasing the rate of

water and SO_2 penetration into the corn kernel. However, it is surprising to know that only Powel et al. (1975) studied the possibilities of breaking corn kernels before steeping. They removed the hull and germ from the corn kernels by dry milling prior to steeping. Although the complete results of the study were not reported they have pointed out some findings and advantages of the process.

1). The steeping time could be reduced from the usual 40-48 hours down to 1/2 -16 hours.

2). The two stages of degermination mills are entirely eliminated.

3). The germ separators or germ hydrocyclones and the machines normally required for germ washing, de-watering and drying are entirely eliminated.

4). The use of multi-stage coarse fiber washing screens becomes unnecessary.

5). The quantity of soluble solids introduced to the corn wet milling process is substantially reduced and hence the cost of separating and drying the solubles is also reduced.

MATERIALS AND METHODS

Sample Preparation :

Table 1 shows the analytical composition of the whole corn kernels (Dekalb 636) used in the study. The starch and protein contents of whole corn kernels were 73.58 and 8.98% (d.b.), respectively. The corn was cleaned using a Kice, Model 6F6, grain cleaner. The cleaned corn (approximately 150 Kg) was degerminated by adjusting the

Table 1 : Composition of Whole Corn. Kernels (M.C. = 11.86%)

Component	Percentage (d.b.)
Starch	73.58
Crude protein	8.98
Crude Fiber	2.04
Oil (Either Extract)	4.12
Ash	1.41

moisture content to about 21% by adding water in several steps. At first, about 5500 ml of water was mixed with corn in a Wenger Mixer to increase the moisture content to about 15%. About 6000 ml of water was added and mixed in the same mixer after overnight tempering. The mixing

procedure was repeated adding 4000 and 2000 ml of water after 1 hour and 1/2 hour periods, respectively. The corn was degerminated using a No. 0 Beall Degerminator to breakout the hulls and germs. The hulls were separated by aspiration. A gravity table, Eriez H1-V1 Model F, was used to separate germs from grits. The flaking grits and other types of grits were dried separately in a continuous flow type dryer (Aeroglide Model C1-12-6-RS). The flaking grits were not used in the experiment since they have a higher market value. The grits were sized to pass through a No. 5 screen and be retained on a No. 10 screen using a Forsberg Dry Granular Separator (Model 2-18-24).

Particle size distribution and mean particle size of corn grits, determined using standard Tyler sieves according to the procedure of Pfost (1976), are given in Table 2 and Appendix A1. About 75% of corn grits (by weight) passed through a Tyler No. 5 screen but over a No. 7 screen (Fig 1). The average particle size of grits was found to be 3319 microns (Appendix A2).

Sixteen representative grit samples of each 1000 grams at 11.2% moisture content were prepared using a Bourner Divider. The moisture content was determined in triplicates (40 g) by the standard air oven method (105 ^{o}C, 72 hr). Standard laboratory analytical procedures according to

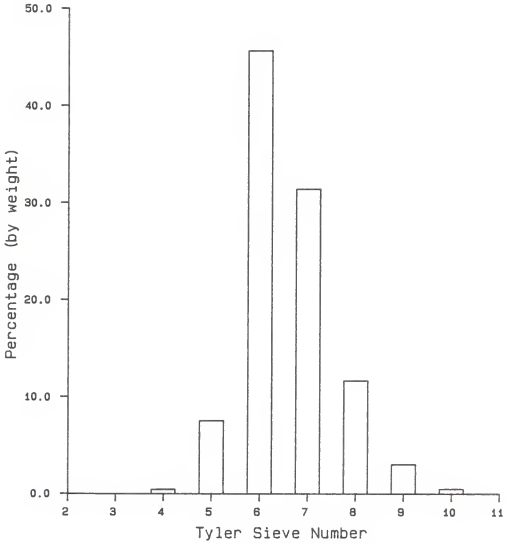

Figure 1 : Particle Size Distribution of Corn Grits

Table 2 : Particle Size Distribution of Corn Grits.

Tyler Sieve No.	Percentage (by weight)
>4	0.44
5	7.53
6	45.55
7	31.34
8	11.65
9	3.02
<9	0.48

AACC (1984) were followed to determine gluten (C.P.), fiber and oil contents. The results of chemical analysis of corn grits are shown in Table 3. Yellow Springs Instrument, Model 27, was used to determine starch contents according to the method describe by Budke (1982). Two representative samples of whole corn were wet milled according to the standard laboratory procedure used by Anderson (1963) to find starch, gluten, course fiber, fine fiber and germ yields.

Wet Milling Procedure

Concentrated sulfur dioxide solution was prepared by bubbling SO_2 gas through distilled water. The SO_2 content

of the solution was determined using an idometric titration procedure similar to that described by Eckhoff and Okos (1983). The solution was diluted with distilled water to get the desired concentration of SO_2 in the steep water. The total amount of diluted SO_2 solution used for steeping of each sample was 2000 ml.

Table 3 : Composition of Corn Grits.
(M.C. = 11.20%)

Component	Percentage (d.b.)
Starch	87.68
Crude protein	8.02
Crude fiber	0.28
Oil (either extract)	1.36
Ash	0.57

The laboratory wet milling procedure used was as described by Anderson (1963). Each sample was steeped in a batch type steeping tank. A water bath temperature control device (Precision Model 291) with a continuous water circulation system was used to maintain a constant temperature of 50 ^{o}C. A Straub Model 4E Grinding Mill was used to finely grind the steeped corn grits. The steeped water was drained off after steeping was completed in the

batch type steeping process of whole corn kernels. However, the steep water was used for grinding corn grits, instead of removing, in the experiment.

A 200-mesh screen was used to separate fiber from the ground slurry. Fiber separation was done manually for 4 hours. The variation of fiber separation among treatments was minimized by employing the same person for all treatments. Specific gravity of the slurry was adjusted to 1.04 by adding starch before starch separation. Approximately 100 grams of starch was required for this purpose in each sample. The starch separation was done by using a starch table constructed according to the specifications reported by Anderson (1963). The flow rate of the slurry on the starch table was maintained at about 300 ml/min. A Whatman No. 4 filter with a vacuum pump was used to separate gluten from the table overflow water. A fixed amount of distilled water was used in the study. Two liters of water for steeping, 5 liters for fiber washing and 1 liter for gluten washing on the starch table were used in each sample.

The starch, gluten and fiber were dried on separate trays for 24 hours in a oven at 50°C. The moisture contents of these products were determined by drying the samples at 120 °C for 2 and 1/2 hours (Anderson 1963). All

calculations were done on a moisture free basis. The protein content and viscosity of starch were determined. The viscosity of starch was measured (AACC,1984), according to the following procedure, using a Brabender Viskograph-E to determine the extent of starch damaged. A starch solution with 7% dry solids was prepared by mixing 32.2 grams of starch (dry solids basis) with 460 ml of the buffer solution. This solution was heated from 35 ^{o}C to 95 ^{o}C with temperature increase of 1.5 ^{o}C/min in the Brabender Viskograph. The temperature was maintained at 95 ^{o}C for 30 minutes and then cooled down to 50 ^{o}C. The maximum viscosity and the temperature at the maximum viscosity were recorded. Fiber was analyzed for starch and gluten contents. Dry solids content of process water was also determined.

Four levels of steeping time (4, 6, 8 and 10 hours) and 2 levels of SO_2 concentration (0.1 and 0.2%) were used in a replicated factorial experimental design. Analysis of Variance with Duncan Multiple Range Test in SAS was used for data analysis.

RESULT AND DISCUSSION

The results of the standard laboratory wet milling of whole corn kernels are given in Table 4. The average starch and gluten yields are about 65 and 8% (d.b.), respectively. These values are in agreement with the result reported by Anderson (1963). The average starch recovery (based on total starch present in the grain) was about 87%.

Table 4 : Wet Milling Data of Whole Corn Kernels.

Component	Yield (% d.b.) Sample 1	Sample 2	Average
Starch Yield	65.63	64.49	65.06
Recovery*1	86.78	87.94	87.36
Protein Content	0.59	0.54	0.57
Gluten Yield	8.16	7.18	7.67
Protein Content	39.48	40.84	40.16
Fine Fiber	4.77	4.90	4.84
Starch Content	32.62	34.87	33.75
Protein Content	28.48	26.32	27.40
Coarse Fiber	7.39	8.59	7.99
Protein Content	15.12	16.07	15.60
Germ	5.92	5.96	5.94
Process Water Yield of Solids*2	2.46	2.72	2.59
Total Dry Matter Recovery	94.33	93.88	94.09

*1 Based on total starch content.
*2 Based on total dry matter content in each sample.

The total average dry matter recovery of wet milling of whole corn kernels found to be about 94%.

The moisture content, bone dry weight and protein content of starch obtained from corn grits for each sample are given in Appendix A3. The average starch yield and recovery of corn grits at different SO_2 concentrations and steeping time periods are shown in Table 5. The highest starch yield of 77.9% (d.b.) was obtained from the corn

Table 5 : Yield of Starch from Wet Milling of Corn Grits.

Steeping Time (hr)	SO_2 Con. (%)	Starch Yield*1 (% d.b.)	Starch Recovery*2 (% d.b.)
4	0.1	76.76 b	87.34 d
6	0.1	77.25 ab	88.06 bcd
8	0.1	77.64 a	88.54 ab
10	0.1	77.89 a	88.82 a
4	0.2	76.88 b	87.50 cd
6	0.2	77.39 ab	88.22 abc
8	0.2	77.83 a	88.76 ab
10	0.2	77.90 a	88.85 a
Control*3	0.2	65.06 c	87.36 d

* Values with the same letters are not significantly different

*1 Duncan Critical Range : 2 = 0.65, 3 = 0.68 4 = 0.70.

*2 Duncan Critical Range : 2 = 0.69, 3 = 0.72, 4 = 0.74.

*3 Wet milling of whole corn kernels.

grits steeped at 0.2% SO_2 concentration for 10 hours.

There was no significant difference in starch yield between the 8 and 10 hours steeping times at 0.1 or 0.2% SO_2 concentrations. The steeping time and SO_2 concentration could be reduced to 8 hours and 0.1%, respectively without any significant decrease in starch yield. The starch yield increased with increasing steeping time for both the 0.1 and 0.2% SO_2 concentrations (Fig 2).

The lowest starch yields of 76.76 and 76.88% (d.b.) were recorded when steeping times were 4 hours and SO_2 concentrations were 0.1 and 0.2%, respectively. There was no significant difference in starch yield in these two treatments.

The average starch recovery from a laboratory wet milling of whole kernels, as reported by Anderson (1963) and the results of this study (Table 4) were 87.90 and 87.36% (d.b.) respectively. The starch recovery from corn grits processed in this study was higher than the above values at all steeping times except for the 4-hour treatment for both the 0.1 and 0.2% SO_2 concentrations. It was found that the starch recovery from corn grits could be increased to 88.85% (d.b.) when steeping time was 10 hours at 0.2% SO_2 concentration (Fig 3). Sulfur dioxide is in direct contact with endosperm proteins during steeping of corn grits. This

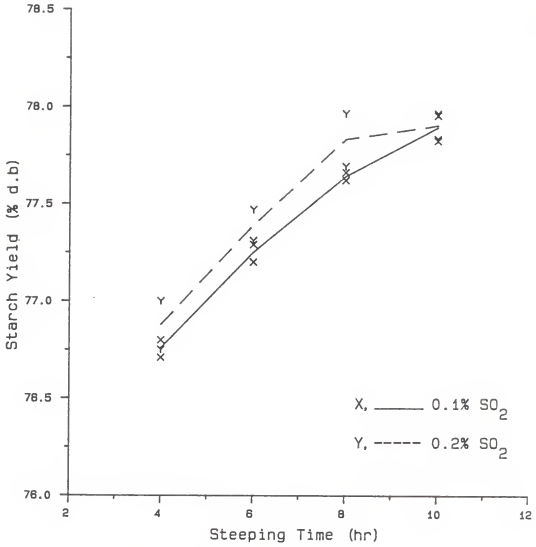

Figure 2 : Starch Yield of Corn Grits

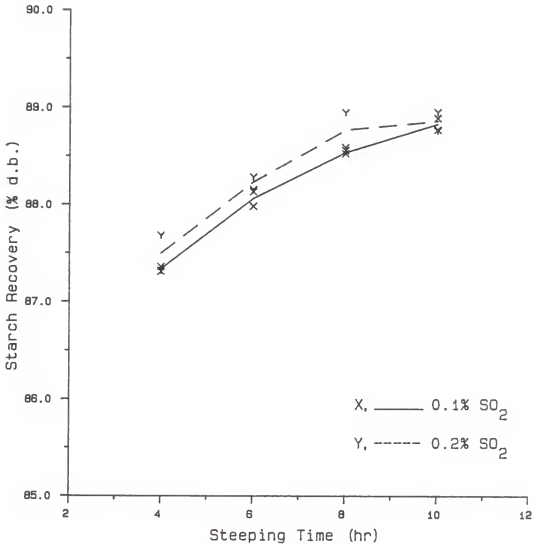

Figure 3 : Starch Recovery of Corn Grits

will increase the rate of protein disintegration even at low SO_2 concentrations.

The effect of SO_2 concentration on starch yield is compared in Table 6. It shows that there was no significant effect of SO_2 concentration (0.1 or 0.2%) on starch yield when the steeping time was between 4 and 10 hours.

Table 6 : Effect of SO_2 Concentration on Starch Yield.

SO_2 Concentration (%)	Average Starch Yield (% d.b.)*
0.1	77.39 a
0.2	77.50 a

* Values with same letters are not significantly different (Duncan Critical Range : 2 = 0.13).

Table 7 shows the effect of steeping time on starch yield. A significant increase in starch yield resulted when steeping time increased from 4 to 6 hours and 6 to 8 hours. No significant increase in starch yield was observed when steeping time increased from 8 to 10 hours.

The degree of purity of corn starch is one of the most important factors which determine the market value. The protein content of starch is, however, used as a measure of

Table 7 : Effect of Steeping Time on Starch Yield.

Steeping Time (hr)	Average Starch Yield (% d.b.)*
4	76.82 a
6	77.32 b
8	77.74 c
10	77.90 c

* Values with same letters are not significantly different.
(Duncan Critical Range : 2 = 0.19, 3 = 0.20).

purity of starch. The starch with the lowest possible protein content is considered as better quality product in the wet milling industry. The protein contents of starch in different treatments are shown in Table 8.

The lowest protein content of 0.49% (d.b.) was found in the starch obtained from the treatment with 10 hours steeping at 0.2% SO_2 concentration. The highest protein contents of 0.73 and 0.70% (d.b.) were observed in the treatments with 4 hours steeping at 0.1 and 0.2% SO_2 concentrations, respectively. The difference in protein content of these two treatments was not significant. It was also found that the protein contents of starch obtained from 6, 8 and 10 hours of steeping were not significantly different for both 0.1 and 0.2% SO_2 treatments. The protein

contents of these treatments was about the same as the protein content of starch obtained from wet milling of whole kernels (Table 4). As far as the protein content (or purity) of the starch is concerned, the steeping time and

Table 8 : Protein Content of Starch Obtained from Wet Milling of Corn Grits.

Steeping Time (hr)	SO_2 Con. (%)	Protein Content (% d.b.)*1
4	0.1	0.73 a
6	0.1	0.54 b
8	0.1	0.51 b
10	0.1	0.50 b
4	0.2	0.70 a
6	0.2	0.55 b
8	0.2	0.50 b
10	0.2	0.49 b
Control*2	0.2	0.58 b

*1 Values with same letters are not significantly different (Duncan Critical Range : 2=0.09, 3=0.10).
*2 Wet Milling of Whole Corn Kernels.

SO_2 concentration could be reduced to 6 hours and 0.1%, respectively, without any significant difference. However, Fig. 4 indicates that protein content of starch decreases

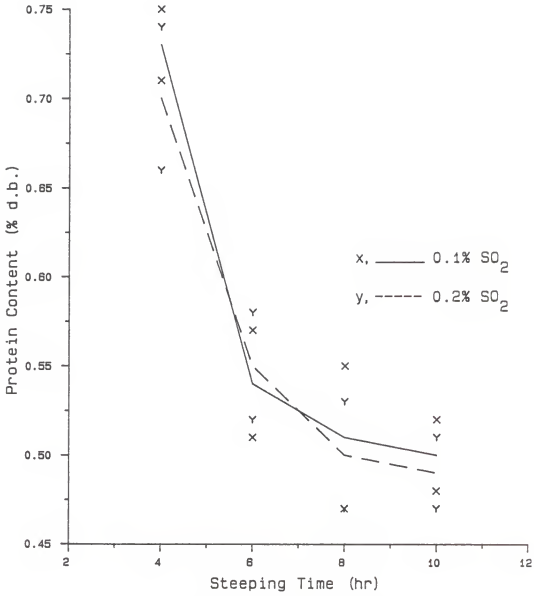

Figure 4 : Protein Content of Starch from Corn Grits

with increasing steeping time.

Protein contents of starch at different steeping time periods are given in Table 9. It shows that a marked decrease in protein content, about 23%, was achieved when steeping time increased from 4 to 6 hours. No significant decrease in protein content of starch was observed when steeping time increase from 6 to 10 hours.

Table 9 : Effect of Steeping Time on Protein Content of Starch.

Steeping Time (hr)	Average Protein Content of Starch (% d.b.)*1	Decrease in Protein Content (%)*2
4	0.72 a	
6	0.55 b	23.61
8	0.51 b	7.27
10	0.50 b	1.96

*1 Values with same letters are not significantly different. (Duncan Critical Range : 2=0.07).
*2 Percentage decrease based on value for preceding time period.

Table 10 compares the effect of SO_2 concentration on protein content of starch. It shows that protein content of starch was not significantly different at the 0.1 and 0.2% SO_2 concentrations when steeping time was between 4 and 10 hours.

Starch separation is usually done on a standard starch table in laboratory wet milling processes. According to Anderson (1963), the average protein content of starch was 0.54% in this process. Two-step continuous centrifuges are, however, used in the wet milling industry for the same purpose. Starch with a protein content as low as 0.3% can

Table 10 : Effect of SO_2 Concentration on Protein Content of Starch.

SO_2 Concentration (%)	Average Protein Content of Starch (% d.b.)*
0.1	0.57 a
0.2	0.56 a

* Values with same letters are not significantly different (Duncan Critical Range : 2=0.05).

be obtained in this process. It is important, therefore, that starch yield be compared at a constant protein content. The starch yield at 0.5% protein content was selected for comparisons.

The starch yields that could have been obtained if the protein contents of the starch were 0.5% are given in Table 11. The highest starch yield of 77.90% (d.b.) was recorded for the treatment with 0.2% SO_2 concentration and 10 hours steeping time. However, there was no significant

difference in starch yield at 0.1 and 0.2% SO_2 concentrations when steeping times were 8 or 10 hours. The data for starch yield at 0.5% protein content are shown in Fig. 5. It shows that the starch yield at 0.2% SO_2 concentration is higher than that of 0.1% for all steeping time levels used in the study.

Table 11 : Yield of Starch if Protein Content were 0.5%.

Steeping Time (hr)	SO_2 Con. (%)	Starch Yield (% d.b.)*1
4	0.1	76.58 c
6	0.1	77.21 abc
8	0.1	77.63 a
10	0.1	77.88 a
4	0.2	76.72 bc
6	0.2	77.35 a
8	0.2	77.82 a
10	0.2	77.90 a
Control*2	0.2	65.02 d

*1 Values with same letters are not significantly different (Duncan Critical Range : 2 = 0.65, 3 = 0.68, 4 = 0.70, 5 = 0.71).
*2 Wet milling of Whole corn kernels.

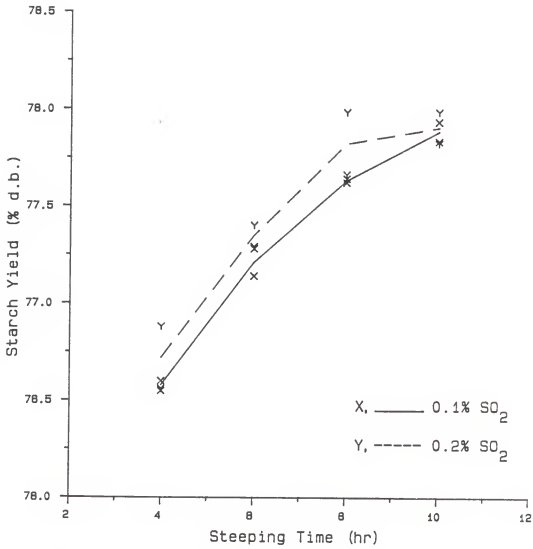

Figure 5 : Starch Yield if Protein Content were 0.5%.

Table 12 shows there was no significant difference between 0.1 and 0.2% SO_2 concentrations on average starch yield even if the starch yields were calculated based on 0.5% protein content. This confirms the conclusion that there was no significant effect between the 0.1 and 0.2% SO_2 concentration on starch yield when steeping time was between 4 and 10 hours.

Table 12 : Effect of SO_2 Concentration on Starch Yield if Protein Content were 0.5%.

SO_2 Concentration (%)	Average Starch Yield (% d.b.)*
0.1	77.33 a
0.2	77.45 a

* Values with same letters are not significantly different (Duncan Critical Range : 2 = 0.15).

The average starch yield that could have been obtained for different steeping time periods if protein contents were 0.5% (d.b.) is given in Table 13. It shows that the highest average starch yield of 77.89% (d.b.) was recorded for the treatments with 10 hours steeping time. This value was not significantly different from that of treatments with 8 hours steeping, but was significantly different from that of treatments with 6 hours steeping. These results are not

different from the starch yields shown in Table 7 calculated according to the actual protein content.

Corn starch is converted into various products such as corn syrups, alcohol, acids, drugs and pharmaceuticals, products of baking industry, etc. in industry. The degree of starch damaged affects the process and/or quality of these end products. However, starch with some starch damage may have some favorable effect on certain production

Table 13 : Effect of Steeping Time on Starch Yield if Protein Content were 0.5%.

Steeping Time (hr)	Average Starch Yield (% d.b.)*
4	76.65 a
6	77.28 b
8	77.73 c
10	77.89 c

* Values with same letters are not significantly different.
(Duncan Critical Range 2 = 0.21, 3 = 0.22).

processes such as corn syrup and alcohol production. The viscosity of starch is considered as an indirect measurement of starch damaged. The lower the viscosity the higher the starch damaged is considered to be.

The viscosity of starch obtained from the different treatments are shown in Table 14 and Appendix A4. These tables show that the highest viscosity (lowest starch damaged) of 490 B.U. was recorded for the starch obtained from the control (starch from wet milling of whole corn kernels) sample. The viscosity of starch from the sample with 0.1% SO_2 and 4 hours of steeping was found to be 462 B.U. This was the highest viscosity among treatments. It was found that a significant decrease in viscosity occurred when steeping time increased from 4 to 6 hours at 0.1% SO_2 concentration (Fig. 6). Fig. 6 graphically shows that the decrease in viscosity was greater at 0.1% SO_2 concentration than for the 0.2% concentration when steeping time increase from 4 to 10 hours.

The temperature at maximum viscosity of starch for different treatments is given in Table 14. It shows that the differences in temperature among treatments are not significant. The highest temperature of 93.3 ^{o}C at maximum viscosity was observed for the control sample.

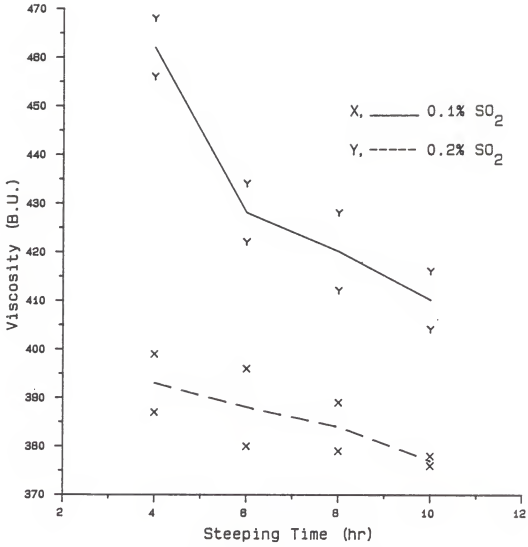

Figure 6 : Viscosity of Starch Obtained from Corn Grits

Table 14 : Viscosity of Starch Obtained from Wet Milling of Corn Grits.

Steeping Time (hr)	SO_2 Con. (%)	Viscosity (B.U.)*1	Temperature at Maximum Viscosity (^oC)*2
4	0.1	462 a	92.3 bc
6	0.1	428 b	92.7 abc
8	0.1	420 bc	92.2 bc
10	0.1	410 cd	92.6 bc
4	0.2	393 cde	92.3 bc
6	0.2	388 de	92.8 ab
8	0.2	384 e	92.2 bc
10	0.2	377 e	92.7 abc
Control Sample*3		490 f	93.3 a

* Values with same letters are not significantly different
*1 Duncan Critical Range : 2 = 24.25, 3 = 25.33, 4 = 26.00.
*2 Duncan Critical Range : 2 = 0.58, 3 = 0.60, 4 = 0.62.
*3 Starch from wet milling of whole corn kernels.

Table 15 compares the effect of SO_2 concentration on starch viscosity. It shows that SO_2 concentration has a great effect on starch viscosity. The difference in average viscosity of starch was about 44 B.U. (Duncan Critical Range = 10.32) which was significant at 5% significant level.

Table 15 : Effect of SO_2 Concentration on Viscosity of Starch.

SO_2 Concentration (%)	Average Starch Viscosity (B.U.)*
0.1	430.0 a
0.2	385.4 b

* Values with same letters are not significantly different (Duncan Critical Range : 2 = 10.32.

The effect of steeping time on starch viscosity is given in Table 16. It shows that the highest average viscosity (lowest damaged) of 428 B.U. was recorded for 4 hours steeping as compared with 394 B.U. for 10 hours steeping. The differences in viscosity among 6, 8 and 10 hours steeping were not significant. However, Fig. 6 shows that the viscosity of starch decreases with increasing steeping time at 0.1 and 0.2% SO_2 concentrations. It was also found that the decrease in viscosity with the increasing steeping time was higher at 0.1% SO_2 concentration than that of 0.2% SO_2 concentration.

The whole corn kernels are steeped in sulfurous acid for certain period of time in the common corn wet milling process. The sulfurous acid gradually penetrates into the kernel with time. The penetrated sulfurous acid (bisulfite iron) reacts with disulfide bonds in the protein matrix of

Table 16 : Effect of Steeping time on Viscosity of Starch.

Steeping Time (hr)	Average Starch Viscosity (B.U.)*
4	428 a
6	408 b
8	401 b
10	394 b

* Values with same letters are not significantly different (Duncan Critical Range : 2 = 14.59.

the corn endosperm. Thus, the direct contact of sulfurous acid with starch would be lower in steeping of whole corn kernels than corn grits. This could be one reason for higher damage of starch obtained from corn grits.

The germs and hulls of corn kernels used in this study were removed by dry milling before steeping. It would be possible that some starch granules may have been damaged during dry milling. These granules were in direct contact with sulfurous acid during steeping of corn grits. This could be a reason for lower viscosity (higher damaged) of starch obtained from wet milling of corn grits. The highest starch damage at the higher concentration of SO_2 (0.2%) supports this conclusion. However, it was not clear whether the dry milling procedure or the steeping process caused the

most starch damage. The above problem may be solved by steeping corn grits at low levels of SO_2 concentration and comparing the viscosity of starch. It may require an increase in the steeping time.

The moisture content, bone dry weight and protein content of gluten for each sample are given in Appendix A5. Table 17 shows the average yield of gluten for different levels of treatments. The lowest yield of 10.32% (d.b.) was recorded when SO_2 concentration was 0.2% and steeping time was 10 hours. The gluten yield was not significantly different at 0.1 and 0.2% SO_2 concentrations when steeping times were 8 or 10 hours.

The gluten yield decreased with increasing steeping time and can be seen graphically in Fig. 7. The protein disintegration would have been higher at a longer steeping time; thus, more starch could have been deposited on the starch table during starch separation. In other words, some starch could have been carried out with gluten over the tail end of the starch table at the shorter steeping time due to poor starch and protein separation. This would increase the gluten yield.

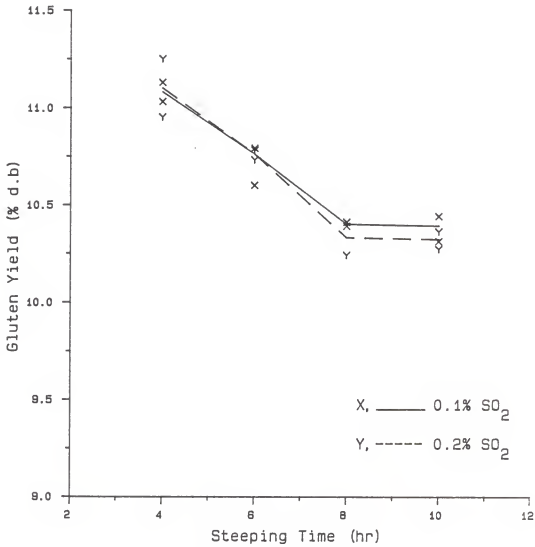

Figure 7 : Gluten Yield of Corn Grits

Table 17 : Yield of Gluten in Wet Milling of Corn Grits.

Steeping Time (hr)	SO_2 Con. (%)	Gluten Yield (% d.b.)*1
4	0.1	11.08 a
6	0.1	10.70 ab
8	0.1	10.40 b
10	0.1	10.38 b
4	0.2	11.10 a
6	0.2	10.76 ab
8	0.2	10.33 b
10	0.2	10.32 b
Control*2	0.2	7.67 c

*1 Values with same letters are not Significantly different (Duncan Critical Range 2 = 0.57, 3 = 0.6, 4=0.61).

*2 Wet Milling of Whole Corn Kernels.

The highest protein content of 39.60% (d.b.) in gluten was observed when SO_2 concentration was 0.2% and steeping time was 10 hours (Table 18). This protein content was not significantly different from that of gluten obtained from the treatment with 0.1% SO_2 concentration at 10 hours steeping. The lowest values of 37.03 and 37.15% (d.b.) protein contents were observed when steeping time was 4 hours and SO_2 concentrations were 0.2 and 0.1%, respectively. It was also found that the protein content of

gluten increase with increasing steeping time (Fig. 8).

Table 18 : Protein Content of Gluten

Steeping Time (hr)	SO_2 Con. (%)	Protein Content (% d.b.)*1
4	0.1	37.15 de
6	0.1	37.89 cde
8	0.1	38.85 bc
10	0.1	39.40 ab
4	0.2	37.03 e
6	0.2	38.01 cde
8	0.2	38.38 bcd
10	0.2	39.60 ab
Control*2	0.2	40.16 a

*1 Values with same letters are not significantly different (Duncan Critical Range : 2 = 1.20, 3 = 1.26, 4 = 1.29, 5 = 1.31).
*2 Wet Milling of Whole Corn Kernels.

As far as fiber yield is concerned, the main objective is to obtain the lowest possible yield yet to have low fiber content in the starch fraction. The lower the fiber yield the higher the starch yield will be. The moisture content, bone dry weight and starch content of fiber for each sample are given in Appendix A6. The average yield of fiber in different treatments is shown in Table 19. These

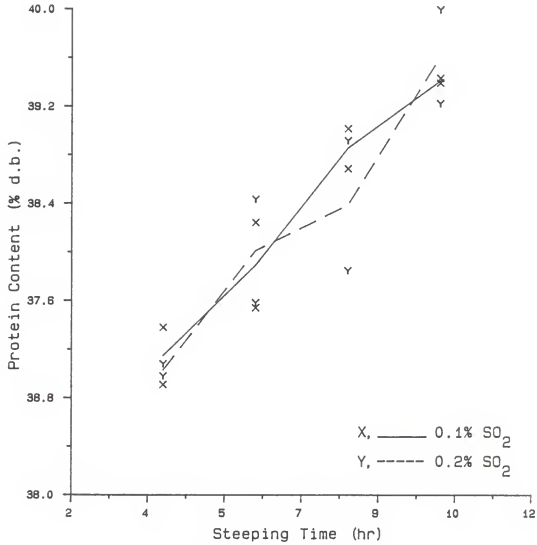

Figure 8 : Protein Content of Gluten

tables show that the lowest fiber yield of 6.30% (d.b.) was recorded when SO_2 concentration and steeping time were 0.2% and 10 hours, respectively. However, the differences in fiber yields were not significant at 0.1 and 0.2% SO_2 concentrations when steeping time was between 4 and 10 hours.

Table 19 : Yield of Fiber in Wet Milling of Corn Grits.

Steeping Time (hr)	SO_2 Con. (%)	Fiber Yield (% d.b.)*1
4	0.1	6.74 a
6	0.1	6.57 a
8	0.1	6.33 a
10	0.1	6.31 a
4	0.2	6.59 a
6	0.2	6.43 a
8	0.2	6.34 a
10	0.2	6.30 a
Control*2	0.2	12.83 b

*1 Values with same letters are not significantly different different
(Duncan Critical Range : 2 = 0.71).
*2 Wet milling of whole corn kernels. Both coarse and fine fiber yields.

The highest fiber yield of 6.74% (d.b.) was observed in the treatment with 0.1% SO_2 and 4 hours steeping.

Fig. 9 shows that the fiber yield decreased with increasing steeping time. It also shows that the fiber yield was higher in the treatment with 0.1% SO_2 than that of the treatment with 0.2% SO_2 content when steeping time was below 8 hours. The maximum difference in fiber yields among treatments was only 0.44% (d.b.).

Starch content of fiber is given in Table 20. It shows that there was no significant different in starch content of fiber in the different treatments. This indicate that SO_2

Table 20 : Starch Content of Fiber

Steeping Time (hr)	SO_2 Con. (%)	Starch Content (% d.b.)*1
4	0.1	28.97 a
6	0.1	28.68 a
8	0.1	28.54 a
10	0.1	28.61 a
4	0.2	29.02 a
6	0.2	28.37 a
8	0.2	28.55 a
10	0.2	28.07 a
Control*2	0.2	33.75 b

*1 Values with same letters are not significantly different (Duncan Critical Range : 2 = 2.22, 3 = 2.32).
*2 Wet milling of whole corn kernels (Starch content of fine fiber fraction).

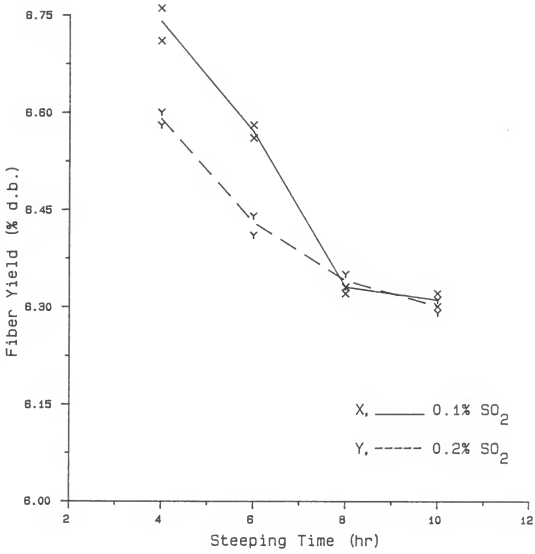

Figure 9 : Fiber Yield of Corn Grits

concentration and steeping time could be reduced to 0.1% and 4 hours, respectively in wet milling of corn grits without any significant loss of starch with fiber. Fig. 10 shows that the average starch content of fiber was about 28.5%. It was also observed the fiber separation was equally easy in all treatments regardless of SO_2 concentrations or steeping times used in the study.

Solids content of process water, as a percentage of total dry matter used in each sample, is given in Table 21. It shows that the solids content of the process water remained constant regardless of SO_2 concentration and steeping time combinations used in the study. The yield of process water in wet milling of corn grits compared well with that of wet milling of whole corn kernels (Table 4). Therefore, it can be concluded that SO_2 concentration, steeping time and type of corn (whole kernels or grits) used in the study have no significant effect on solids content of the process water. The average solids content of processed water was about 2.6% (Fig. 11).

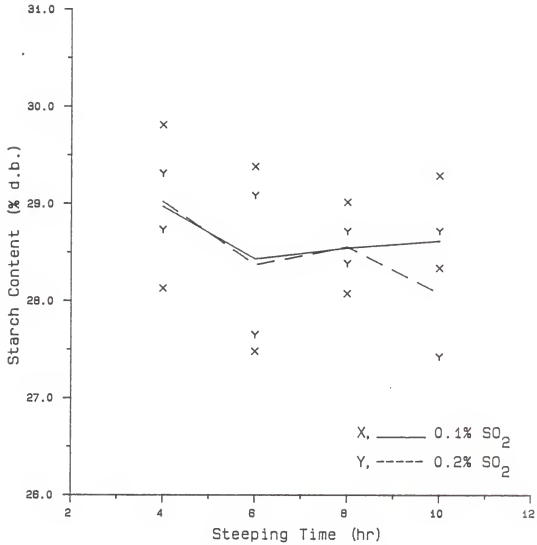

Figure 10 : Starch Content of Fiber

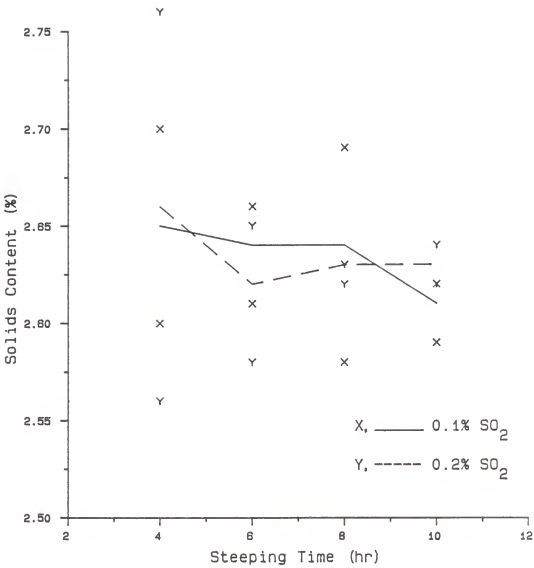

Figure 11 : Dry Solids Yield of Process Water

Table 21 : Yield of Solids in Process Water in Wet Milling of Corn Grits.

Steeping Time (hr)	SO_2 Con. (%)	Process Water		
		Volume (ml)	Weight of Solids (g)	Solids Yield (% d.b.)*1
4	0.1	6930	23.57	2.65 a
6	0.1	6905	23.46	2.64 a
8	0.1	7095	23.41	2.64 a
10	0.1	7030	23.18	2.61 a
4	0.2	6965	23.68	2.66 a
6	0.2	6840	23.23	2.62 a
8	0.2	6775	23.36	2.63 a
10	0.2	6780	23.39	2.63 a
Control*2	0.2	9850	34.24	2.59 a

* Values with same letters are not significantly different (Duncan Critical Range : 2 = 0.15).
*1 As a percentage of total dry matter used in each sample.
*2 Wet milling of Whole corn kernels.

The total dry matter recovery of the treatments are given in Table 22 and Appendix A7. It was found that the SO_2 concentration (0.1 or 0.2%) or steeping time (4,6,8 or 10 hours) had no significant effect on total dry matter recovery. It was also found that about 97% dry matter could be recovered from wet milling of corn grits (Fig 12). The average total dry matter recovery from wet milling of whole corn kernels was 94.09% (d.b.). The dry

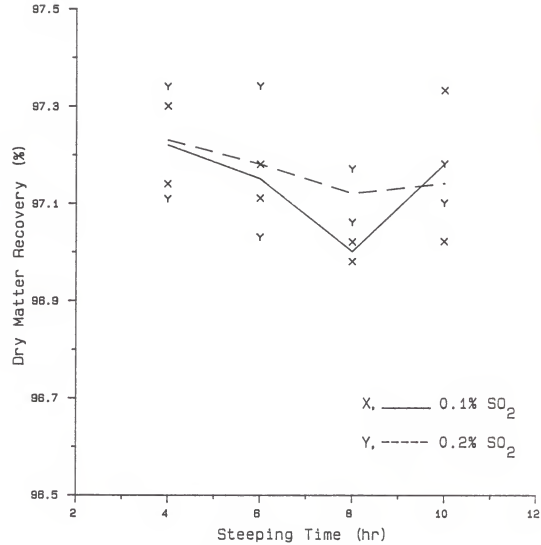

Figure 12 : Total Dry Matter Recovery of Corn Grits

Table 22 : Total Dry Matter Recovery from Wet Milling of Corn Grits.

Steeping Time (hr)	SO_2 Con. (%)	Total D.M. Recovery*1 (% d.b.)
4	0.1	97.22 a
6	0.1	97.15 a
8	0.1	97.00 a
10	0.1	97.18 a
4	0.2	97.23 a
6	0.2	97.19 a
8	0.2	97.12 a
10	0.2	97.14 a
Control*2	0.2	94.09 b

*1 Values with same letters are not significantly different (Duncan Critical Range : 2 = 0.39)
*2 Wet milling of whole corn kernels.

matter recovery of corn grits could be considered as higher recovery when compared with that of wet milling of whole corn kernels. Anderson (1963) reported that if care is taken, 95 - 97% of the total dry solids could be recovered from a laboratory wet milling process.

CONCLUSIONS

1). The steeping time and SO_2 concentration could be reduced to 0.1% and 8 hours respectively in wet milling of corn grits without any significant different in starch yield or protein content of starch.

2). As high as 88.85% (d.b.) starch recovery could be achieved in wet milling of corn grits. The starch recovery of wet milling of whole corn kernels was 87.36% (d.b.).

3). Protein content of starch in the study compared well with that of starch obtained from whole corn kernels.

4). The SO_2 concentrations of 0.1 or 0.2% had no significant effect on average starch yield, protein content of starch, gluten yield, starch content of fiber, yield of process water and total dry matter recovery for the range of 4 to 10 hours of steeping times.

5). The viscosity of starch obtained from corn grits was significantly lower than that of starch from whole corn kernels. The highest and lowest viscosity (lowest and highest starch damaged) among treatments were 462 and 377 B.U., respectively. The 0.1% SO_2 concentration caused less starch damaged than that of 0.2% SO_2

concentration. The lowest starch damaged was observed in the treatment with 0.1% SO_2 concentration and 4 hours steeping time.

6). The gluten yield decreased with increasing steeping time, however, the protein content of the gluten increased with increasing steeping time.

7). The fiber yield decreased with increasing steeping time. The difference between highest and lowest fiber yield was only 0.41% (d.b.). When steeping time was between 8 and 10 hours, both 0.1 and 0.2% SO_2 concentrations had no significant effect on fiber yield.

8). Solids contents of processed water was not dependent on either SO_2 concentration or steeping time when SO_2 concentration was 0.1 or 0.2% and steeping time was between 4 and 10 hours.

9). The total dry matter recovery of wet milling of corn grits was about 3% (d.b.) higher than that of wet milling of whole corn kernels.

10). Neither SO_2 concentration (0.1 or 0.2%) nor steeping time (4, 6, 8 or 10 hours) had a significant effect on total dry matter recovery.

SUGGESTIONS FOR FURTHER STUDIES

Based on the findings of the study, the following suggestions are made for further studies :

1). It is well known that the size of a particle is one of the most important factors which determine the steeping time. However, only one size range of corn grits were used in the study. An experiment should be conducted using different size of corn grits to find the effect of particle size on steeping.

2). It was found that the difference in effect of 0.1 and 0.2% SO_2 concentrations on average starch yield, protein content of starch, gluten yield, starch content of fiber and solids content of processed water were not significant for a steeping time of between 4 and 10 hours. It was also found that the 0.2% SO_2 concentration cause more starch damaged than that of 0.1% concentration. It would be beneficial to find the possibilities of further reduction of SO_2 concentration in steeping.

3). Kerr (1950) pointed out that the conditions of starch-gluten slurry such as pH, temperature, ironic strength and flow rate of the slurry on the starch table have marked effect on starch separation. There were some

indications to believe that the starch and gluten ratio also has a considerable effect on starch separation in addition to the above conditions. It is, therefore, important that an experiment be conducted to find the effect of starch-gluten ratio on starch separation.

4). Krochta et al. (1980) mentioned the maximum starch yield from wet milling of whole corn kernels could be obtained when the water-corn ratio is 2:1 in steeping. This ratio was used in the study. However, it may possible to reduce this ratio in wet milling of corn grits. A research project to find the effect of the water-corn grits ratio on wet milling qualities of corn grits should be conducted.

5). It was found the starch obtained from wet milling of corn grits had more starch damaged than starch obtained from wet milling of whole corn kernels. It was not clear whether the corn dry milling procedure or steeping process cause the most starch damaged. This should be investigated.

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APPENDIX

Table A1 : Particle Size Distribution of Corn Grits

Tyler Sieve No.	Sample 1	Sample 2	Sample 3	Total Weight (g)	Percentage by Weight
>4	1.58	2.06	1.64	5.28	0.44
5	33.58	26.15	30.49	90.22	7.53
6	187.96	180.95	177.19	546.10	45.55
7	122.44	129.14	124.12	375.70	31.34
8	42.54	47.91	49.22	139.67	11.64
9	10.02	11.66	14.54	36.22	3.02
<9	1.40	1.70	2.62	5.72	0.48
Total	399.52	399.57	399.82	1198.91	100.00

Table A2 : Calculation Procedure to Find Mean Particle Size of Corn Grits.

Screen No.	d_i (u)	W_i (g)	$W_i \text{Log}(d_i)$	$\text{Log}(d_i) - \text{Log } d_{gw}$ (X)	X^2	$W_i * X^2$
3	6730					
4	4760	5.28	19.85	0.235	.055	0.290
5	4000	90.22	328.40	0.115	0.013	1.173
6	3360	546.10	1949.58	0.045	0.002	1.092
7	2830	375.70	1311.19	-0.035	0.001	0.376
8	2380	139.67	477.67	-0.105	0.011	1.536
9	2000	36.22	120.97	-0.185	0.034	1.231
10	1680	5.72	18.65	-0.265	0.070	0.400
		1198.91	4226.31		0.186	6.098

$$\text{Log } d_{gw} = \frac{E(W_i \text{ Log } d_i}{E\ W_i} = \frac{4226.31}{1198.91} = 3.525$$

$$d_{gw} = 3319 \text{ u}$$

$$(\text{Log } S_{gw})^2 = \frac{6.098}{1198.91} = 5.086 \times 10^{-3}$$

$$S_{gw} = 1.178$$

Table A3 : Moisture Content, Bone Dry Weight and Protein Content of Starch Obtained from Wet Milling of Corn Grits.

Steeping Time (hr)	SO_2 Con. (%)	Weight of Starch (g)	M. C. (% w.d)	Born Dry Weight (g)	Protein Content (% d.b.)
4	0.2	719.99	4.94	684.42	0.66
4	0.2	738.76	7.66	682.17	0.74
4	0.1	757.08	9.94	681.83	0.71
4	0.1	740.87	7.87	682.56	0.75
6	0.2	730.00	5.68	688.54	0.58
6	0.2	726.47	5.42	687.10	0.52
6	0.1	725.13	5.38	686.12	0.57
6	0.1	731.78	6.13	686.92	0.51
8	0.2	733.25	5.50	692.92	0.47
8	0.2	740.73	6.78	690.51	0.53
8	0.1	740.33	6.77	690.21	0.55
8	0.1	737.16	6.41	689.91	0.47
10	0.2	736.89	6.13	691.72	0.51
10	0.2	732.56	5.41	692.93	0.47
10	0.1	738.97	6.25	692.78	0.52
10	0.1	730.94	5.38	691.62	0.48

Table A4 : Viscosity of Starch Obtained from Wet Milling of Corn Grits and Whole Kernels.

Steeping Time (hr)	SO_2 Con. (%)	Maximum Viscosity (B.U.)	Temperature At Maximum Viscosity (^oC)
4	0.2	399	92.5
4	0.2	387	92.1
4	0.1	468	92.2
4	0.1	456	92.3
6	0.2	380	92.9
6	0.2	396	92.7
6	0.1	434	92.9
6	0.1	422	92.5
8	0.2	379	92.0
8	0.2	389	92.3
8	0.1	428	92.1
8	0.1	412	92.3
10	0.2	376	92.8
10	0.2	378	92.6
10	0.1	416	92.7
10	0.1	404	92.5
Control*	0.2	494	93.5
Control*	0.2	486	93.0

* Starch from wet milling of whole corn kernels.

Table A5 : Moisture Content, Bone Dry Weight and Protein Content of Gluten from Wet Milling of Corn Grits.

Steeping Time (hr)	SO2 Con. (%)	Weight of of Gluten (g)	M. C. (% w.d)	Born Dry Weight (g)	Protein Content (% d.b.)
4	0.2	101.03	3.64	97.35	36.98
4	0.2	106.45	6.11	99.95	37.08
4	0.1	103.46	4.38	98.93	37.38
4	0.1	103.36	5.12	98.07	36.91
6	0.2	101.00	5.16	95.79	37.58
6	0.2	99.76	4.37	95.40	38.43
6	0.1	101.65	5.68	95.88	38.24
6	0.1	98.83	4.68	94.20	37.54
8	0.2	95.44	4.66	90.99	37.84
8	0.2	98.13	5.72	92.52	38.91
8	0.1	96.95	4.56	92.53	39.01
8	0.1	97.85	5.61	92.36	38.68
10	0.2	97.34	5.38	92.10	39.98
10	0.2	95.30	4.26	91.24	39.21
10	0.1	99.14	6.44	92.76	39.38
10	0.1	96.75	5.30	91.62	39.42

Table A6 : Moisture Content, Bone Dry Weight and Starch Content of Fiber from Wet Milling of Corn Grits.

Steeping Time (hr)	SO2 Con. (%)	Weight of of Fiber (g)	M. C. (% w.d)	Born Dry Weight (g)	Starch Content (% d.b.)
4	0.2	62.73	6.54	58.63	29.31
4	0.2	62.79	6.91	58.45	28.73
4	0.1	64.98	7.58	60.05	28.13
4	0.1	63.78	6.54	59.61	29.81
6	0.2	60.67	5.72	57.20	27.65
6	0.2	61.01	6.61	56.98	29.08
6	0.1	62.59	6.61	58.45	27.98
6	0.1	61.53	5.28	58.28	29.38
8	0.2	60.59	6.92	56.40	28.38
8	0.2	60.90	7.58	56.28	28.71
8	0.1	59.49	5.36	56.30	29.01
8	0.1	59.24	5.17	56.18	28.07
10	0.2	59.16	5.54	55.88	28.71
10	0.2	60.17	6.79	56.08	27.42
10	0.1	59.31	5.28	56.18	29.08
10	0.1	59.73	6.21	56.02	28.13

Table A7 : Total Dry Matter Recovery of Wet MIlling of Corn Grits and Whole Kernels.

Steeping Time (hr)	SO2 Con. (%)	Dry Matter Recovery (% d.b.) Starch	Gluten	Fiber	P.W.*1	Total D.M. Recovery (% d.b.)
4	0.2	77.00	10.95	6.60	2.56	97.11
4	0.2	76.75	11.25	6.58	2.76	97.34
4	0.1	76.71	11.13	6.76	2.70	97.30
4	0.1	76.80	11.03	6.71	2.60	97.14
6	0.2	77.47	10.78	6.44	2.65	97.34
6	0.2	77.31	10.73	6.41	2.58	97.03
6	0.1	77.20	10.79	6.58	2.61	97.18
6	0.1	77.29	10.60	6.56	2.66	97.11
8	0.2	77.96	10.24	6.35	2.62	97.17
8	0.2	77.69	10.41	6.33	2.63	97.06
8	0.1	77.66	10.41	6.33	2.58	96.98
8	0.1	77.62	10.39	6.32	2.69	97.02
10	0.2	77.83	10.36	6.29	2.62	97.10
10	0.2	77.96	10.27	6.31	2.64	97.18
10	0.1	77.95	10.44	6.32	2.62	97.33
10	0.1	77.82	10.31	6.30	2.59	97.02
Control*2		65.63	8.16	18.08*3	2.46	94.33
Control*2		64.49	7.18	19.45	2.72	93.84

*1 Based on total dry matter in each sample.
*2 Wet milling of whole corn kernels.
*3 Dry matter recovery as fine fiber, coarse fiber and germ.

OPTIMUM STEEPING PARAMETERS OF CORN GRITS

by

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B.S., University of Peradeniya, 1981

AN ABSTRACT OF A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

AGRICULTURAL MECHANIZATION

KANSAS STATE UNIVERSITY
Manhattan, Kansas

1988

ABSTRACT

The optimum steeping parameters of corn grits were investigated. Corn grits were obtained from the standard dry milling of whole corn kernels. One size of corn grits (pass through a No. 5 and over a No. 10 screen), 2 levels of SO_2 concentrations (0.1 and 0.2%) and 4 levels of steeping time (4, 6, 8 and 10 hours) were studied. It was found that the steeping time and SO_2 concentration could be reduced to 0.1% and 8 hours respectively without any significant difference in yield or protein content of starch. Starch recovery of as high as 88.85% (d.b.) was achieved from the grits in the study compared to a 87.36% (d.b.) recovery form the control samples (48 hr., 0.2% SO_2, whole kernels). The highest and lowest values recorded for starch viscosity among treatments were 462 and 377 B.U., respectively. Both of these values were significantly lower than viscosity of starch obtained from wet milling of whole kernels which was 490 B.U.

Gluten yield decreased from 11.10 to 10.32% (d.b.) when steeping time increased from 4 to 10 hours at 0.2% SO_2 concentration. The difference between highest and lowest fiber yields among treatments was only 0.41% (d.b.). Sulfur dioxide concentrations and steeping times used in the study had no significant effect on either solids content of the process water or total dry matter recovery. About 3% (d.b.) higher total dry matter recovery was observed in wet milling of corn grits than that of whole corn kernels.