THE INDUSTRIAL RECOVERY OF STARCH FROM SORGHUM GRITS

by

WALTER DROBOT

B. S., Drexel Institute of Technology, 1944

A THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Chemical Engineering

KANSAS STATE COLLEGE
OF AGRICULTURE AND APPLIED SCIENCE

1950

TABLE OF CONTENTS

Introduction 1
Materials and Methods. 3
    Hydraulic Milling 3
    General Procedure 4
    Specific Procedures 19
        Trial Runs 19
        Semi-Continuous Milling. 19
        Interrupted Batch Milling. 23
        Periodic Flushing. 24
    Solids Content. 25
    Starch Product. 26
Data and Results 27
    Trial Runs. 27
    Semi-Continuous Milling 30
    Interrupted Batch Milling 33
    Periodic Flushing 37
    Starch Quality. 44
    Power Consumption 46
Discussion 47
Conclusions. 52
Acknowledgment 54
Bibliography 55

INTRODUCTION

High crop yields of sorghum grain under a wide range of natural conditions recommend this plan for further production in the State of Kansas.

In addition to obvious uses, such as feed material, the sorghum grain is valuable for its chemical components of starch, waxes, fats and fibrous material.

This investigation was carried out for the purpose of determining and testing methods for the efficient recovery of starch from sorghum, with a view toward industrial application, thus increasing the potential value of sorghum crops grown in Kansas.

Barham (2) lists an analysis of sorghum grits taken from seven samples of Westland Milo, as shown in Table 1.

Table 1. A typical analysis of Westland Milo sorghum grits (dry basis).

Component	Percent
Protein	10.13
Ether Extract	0.84
Ash	0.46
Carbohydrates	88.57
Total	100.00
Starch by $CaCl_2$ method	83.26

Johnston (4) investigated the feasibility of recoverying starch from whole sorghum grain using procedures similar to those used in the commercial production of cornstarch. A survey of representative varieties of sorghum grains indicated that starch of satisfactory quality and purity is available in favorable yields from this source. Johnston's method involved the use of two separate steps: A preliminary grinding operation of steeped whole grain using a disc grinder, followed by a final grinding in a Buhr mill, both carried out in the presence of water. Intermediate steps between these two operations were taken to separate the partially ground grain from recovered starch and other components. Immediately before introduction to the Buhr mill, the germ fraction of the sorghum grain containing essentially no starch was withdrawn, leaving only the starch rich portion to be subjected to final grinding. After leaving the Buhr mill, the slurry was separated into two fractions by a screening operation. One of these fractions was bran, fibre and unground endosperm; the other a suspension of starch and gluten in water. The starch was recovered by use of two consecutive semi-continuous settling operations, followed by a final washing and decantation of wash water.

With the development of an efficient dry milling process for producing bran germ and an endosperm fraction, called grits, from Milo sorghum, it became desirable to investigate the production of starch from these grits.

Banowetz (1) studied the recovery of starch from sorghum grits by means of a single step hydraulic milling process in the presence of water. Operating speed, duration of milling and initial sorghum concentration were investigated for their effect on quality and yield of starch. For the experimental conditions studied, a starch of satisfactory quality was produced, but yields, on the basis of whole sorghum grain, were lower than obtained in Johnston's process. His data indicated the possibility of an optimum exposure time of sorghum to the hydraulic milling process.

It was endeavored, in the present work, to produce a starch of high quality in a yield commensurate with that present in the raw material. Recovery was carried out under a wide variety of conditions to determine the effect of operating variables, including some not previously studied, on product yield, quality and industrial feasibility.

MATERIALS AND METHODS

Hydraulic Milling

As in the previous investigation of Banowetz (1), the method of hydraulic milling was used to remove starch from the whole sorghum grits. Sorghum grits, produced from Westland Milo, were used for this and the previous work by Banowetz. Grits are the starchy endosperm part of the grain remaining after the removal of the outer bran coat and the oil bearing germ. This raw material was supplied by Dodge City Industries, Inc., Dodge City, Kansas.

Hydraulic milling utilizes the principle of physical disruption in the presence of water to remove the starch from the grit structure. Mechanical pressure is not applied directly to the grits, as has previously been the case in the method using the Buhr mill. Instead, breakdown is induced by the action of sharpened blades rotating through a slurry of grits and water contained in a shell. Disruption of the grit structure also takes place simultaneously from the turbulence brought about by the blade motion through the grit-water slurry. The grit particles are abraded as they strike the shell wall, strike each other, or undergo exposure to a rapidly moving fluid stream. It is believed that the advantages of this process are lower power consumption and less tendency for local overheating of the slurry.

Two hydraulic shells, designated A and B, were used in this investigation. They were so constructed that each could be mounted on a common base which carried a reinforced shaft on whose length were spaced sharpened blades (Plates I, II and III). Housings carrying bearings supported both ends of the shaft. Experimental runs were carried out at speeds ranging from 2,600 to 4,700 r.p.m. The motive power for the grinding operation was obtained by connecting the blade shaft to a 10 H.P. Fairbanks-Morse induction motor, operating at 1,170 r.p.m., by means of a rubber V-belt. Operating speeds could be selected by varying the ratio of the shaft and motor pulleys.

General Procedure

Prior to milling of sorghum grits, they were exposed for two hours to hot water, under continual mixing, to soften and loosen the grit structure and facilitate the removal of the starch.

EXPLANATION OF PLATE I

Fig. 1. Drawing of mill base.
Fig. 2. Drawing of mill shaft.

PLATE I

MAT'L: MILD STEEL

SCALE: 1"=10"

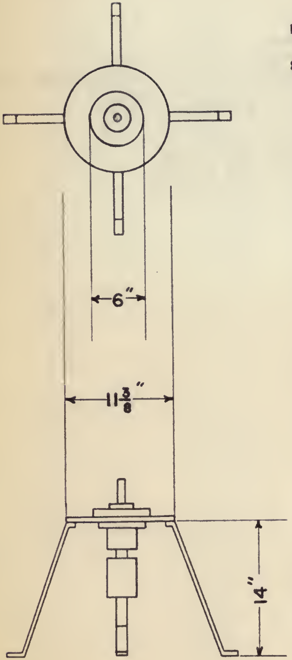


FIG. 1

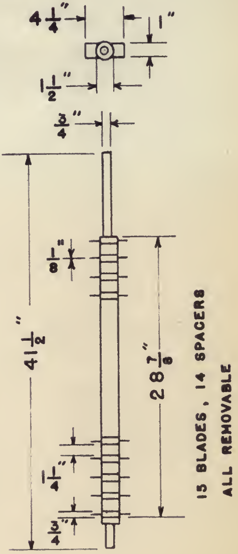
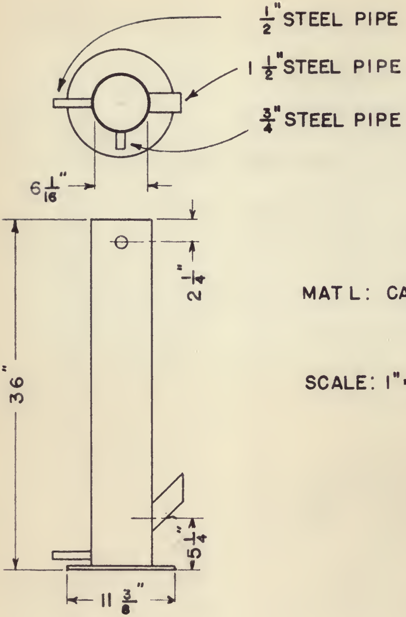


FIG. 2

EXPLANATION OF PLATE II

Drawing of Mill A.

PLATE II



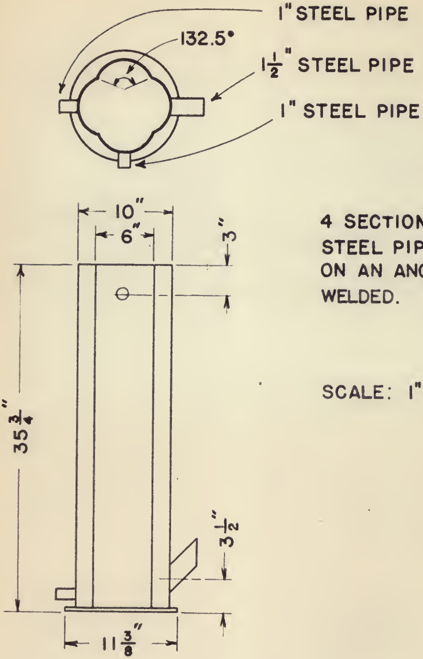
MAT L: CASING — 6" STEEL PIPE

SCALE: 1" = 10"

EXPLANATION OF PLATE III

Drawing of Mill B.

PLATE III



4 SECTIONS 6" STD. STEEL PIPE. EACH CUT ON AN ANGLE OF 132.5°. WELDED.

SCALE: 1" = 10"

This pre-conditioning, known as steeping, was carried out in a cone-bottomed drum fitted with a valve to allow easy escape of the contents from the bottom of the container. Stirring was carried out with a type RA Lightnin Mixer, 4 H.P., operating at approximately 700 r.p.m.

The weighed quantity of raw grits was made up with cold tap water to a volume of 27.5 gallons, and mixing was begun. Heating was accomplished by circulating hot water, from a steam-water heat exchanger, through copper coils in the steep tank. It was endeavored to hold the temperature of the tank contents at 130° F. Approximately 30 minutes' time was required for the batch to reach constant steeping temperature, and this temperature ranged between 128° and 133° F. on any given day.

After two hours had elapsed, the mixer was turned off, and a few minutes were allowed for the grits to settle to the bottom of the steep tank. The grits were then drawn off, with excess steep water, and poured into a bucket with a 40-mesh screen bottom placed above the steep tank. The grits were retained, and the excess steep water flowed back into the steep tank, leaving the steep water available for use in the hydraulic milling operation. This step was repeated three or four times to insure complete separation of the steeped grits. The steeped grits were allowed to drain from 15 minutes to two hours before milling. The steeping operation was carried out in the same way for all experimental runs.

The steeped, drained grits were weighed before introduction into the hydraulic mill. For most tests, the top bearing and cover plate were removed from the mill, and the grits were loaded through this opening. For runs where less than 2.5 pounds of raw grits were used, the steeped, drained grits were placed in the feed hopper and washed into the mill with steep water.

The run was begun by putting the mill into motion and then adding steep water through the feed hopper to fill the mill to capacity. The quantity of steep water introduced was weighed, and the weight per cent of dry grits present (on dry basis) in the mill was calculated. Practically all runs were carried out with the introduced steep water at a temperature of 120° F.

During milling, the operating speed was periodically checked by a tachometer placed on the upper shaft end. The rate of power consumption was also checked periodically by noting the time required for the disc of the wattmeter placed in the motor circuit to make a given number of revolutions. These observations were recorded. To approximate the actual power consumed in separating starch and gluten from the whole grits, it was necessary to subtract the power consumed by the mill while operating with a full capacity of water only. This "no load" power requirement was determined at various shaft speeds.

Upon completion of the milling operation, the slurry, consisting of residual grits, starch and gluten suspended in water, was drained from the mill through the bottom exit valve, and the mill was rinsed with water. The temperature of this slurry after milling usually ranged from 120° to 125° F., depending on the length of time the slurry was subjected to milling. Longer milling periods increased the temperature of the exit slurry. In occasional cases where overheating of the bearings was encountered, the slurry had a final temperature as high as 130° F.

The slurry and rinse water were then transferred to a hopper through which they flowed upon a vibrating, 200-mesh stainless steel screen. The residual grits were retained on the screen, while the aqueous suspension of starch and gluten, called starch milk, was collected in a mixing drum where it was continually stirred to prevent settling of the starch. The apparatus supporting the 200-mesh screen, called the shaker, contained four beds into which were fitted screens of 9½-inch by 33-inch size. The downward slope of these screens could be adjusted to give maximum flow of slurry across the screen while substantial separation of starch milk was taking place. To speed the flow of starch milk from the grits, an oscillating motion of the screens, imparted by an eccentric, kept the slurry in constant motion. The oscillating frequency was 800 times per minute, and the magnitude of horizontal motion was ½ inch. Only one or two screens were used for grit separation, and the downward angle of the screens was approximately 5°.

After separation of the residual grits, they were dried, or, for some runs, returned to the hydraulic mill for further grinding.

Recovery of starch from the extracted starch milk was brought about by a semi-continuous settling operation referred to as tabling. The milk was allowed to flow through a gently inclined trough where the suspended starch particles settle and accumulate on the trough bottom. The remaining suspension containing gluten and other impurities, and perhaps unseparated starch, passed off the trough as effluent. The separation efficiency of the tabling operation has a great influence on the freedom of the starch from the gluten and protein fraction. A relatively rapid flow rate of starch milk through the trough will leave more of the gluten in the effluent liquid, yielding a purer starch product. Simultaneously, some starch will not settle out at the higher flow rates, resulting in a lower yield of product. Conversely, a relatively low flow rate yields more starch of greater impurity. The optimum flow rate depends on the separation efficiency and purity of starch desired. The proper range of flow rates for any given table is influenced by the table slope and width. Once established, the flow rate must remain constant at all times to achieve uniform purity and separation.

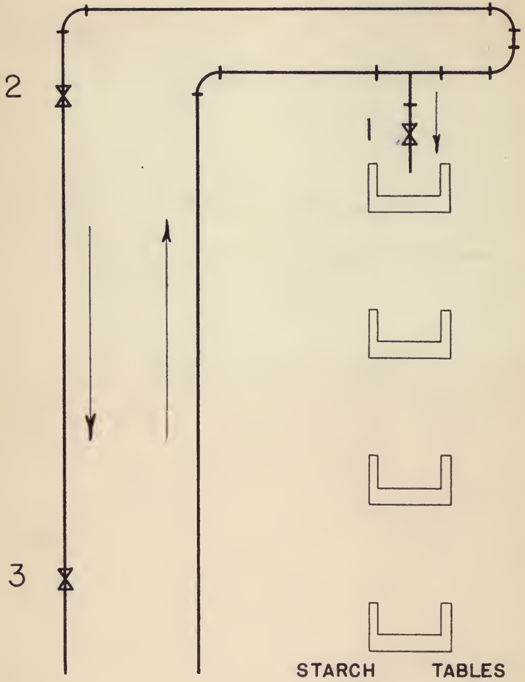
The settling tables used consisted of four sections, one above the other, operated in series. The horizontal length of each section was 30 feet, with a drop of one inch in every 10 feet. The inside of the tables was 5 3/4 and 2 1/2 inches high (Plate 4).

The piping from the milk tank in the main laboratory to the starch tables on the floor above was constructed so that there was a complete circuit of moving fluid from the tank to the tables and back again to the tank. A ½ H.P. bronze gear pump operating

EXPLANATION OF PLATE IV

Schematic diagram of piping to starch tables.

PLATE IV



at 810 r.p.m. moved the starch milk through the lines. This constant motion of the milk prevented settling out of the starch in the lines which would cause clogging and unsteady flow. A portion of this main stream was continually diverted to the table while the remainder was returned to the milk tank.

Preparatory to tabling, exit valve 1 was closed, and throttling valves 2 and 3 were fully opened. Fresh water was then pumped through the system to flush it of residual matter and rust.

After the milk was fully collected, its temperature, volume and specific gravity were measured and noted. Pumping of the milk through the tabling lines was then begun, with valves 1, 2 and 3 set, as previously, for flushing. Tabling was started by opening valve 1 fully and adjusting the flow rate through the tables at 0.6 gal./min. by means of valve 2. Valve 3 was always fully open. Care was taken not to have valve 1 and also 2 or 3 closed simultaneously.

The flow rate was quickly adjusted to 0.6 gal./min. by noting the volume of milk collected in a small calibrated measuring bottle over a period of 15 seconds. Necessary changes were made on throttling valve 2, and a constant flow rate maintained itself until the contents of the milk tank were low enough to allow air in the lines, at which time spurting from the exit line occurred.

For the early runs, a tabling flow rate of 0.5 gal./min. was used, but it was noted that an excessive amount of gluten was deposited on the starch. An increased rate of 0.6 gal./min.

showed great improvement in purity, and this was adopted for the remaining runs.

When tabling of milk was completed, approximately five gallons of fresh water were added to the milk tank and allowed to run on the table to rinse the lines. Approximately 10 gallons of fresh water were then added to the milk tank, and this was introduced to the tables at the same rate of 0.6 gal./min. to wash the collected starch.

In some cases, the effluent liquid was collected for further separation or visual inspection, but, for most runs, it was discarded.

The tabled starch was allowed to drain on the tables for a minimum of two hours, after which it was scraped up. Final traces were collected with a paint brush, insuring practically complete removal.

The final starch product and the residual grits were placed in trays and dried in a Koch air circulating tray drier for eight hours at 155° F. At the end of four hours' drying time, both starch and grit particles were broken up and redistributed over the trays to prevent case-hardening. At the end of eight hours, the trays were removed, weighed immediately, and the yields were noted. It was found that, with the louvres on the sides of the drier open, eight hours' drying time resulted in a substantially constant weight. Batches of starch dried in the oven showed an accumulation of fine particles in the center portion of the tray. Moisture balances and weight tests showed that material losses from circulating air in the drier were negligible.

Specific Procedures

Four general procedures were used to study the effects of process variables on starch yield. Banowetz (1) indicates the possibility that prolonged exposure of previously separated starch to continued milling reduces the product yield. It was in this direction that further tests were carried out.

Trial Runs

Early runs involved the removal of slurry from the top of Mill A and subsequent separation of starch milk on the shaker. The grits were then flushed back into the mill for further grinding, filling the mill again to capacity.

Banowetz (1) used a grit slurry circulation system whereby the grits were withdrawn from the bottom of the mill, passed upward by a pump and returned to the mill through a pipe connection at the top of the mill. This tended to keep the grits in the slurry from stratifying and collecting near the mill bottom. For early runs (Table 3), the direction of this external circulation was reversed so that slurry passed out the top of the mill and was returned to the bottom section. The purpose of this change was to prevent the immediate removal into the circulation system of newly fed grits entering at the bottom of the mill.

Semi-Continuous Milling

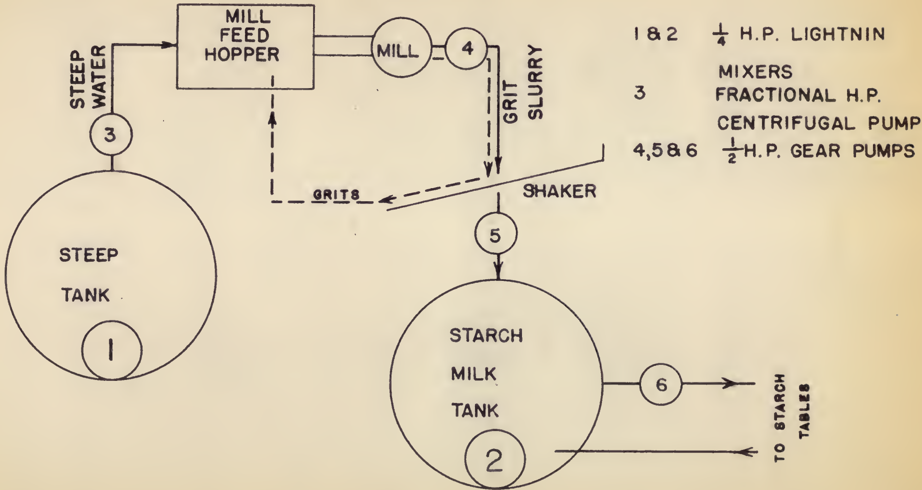
Equipment changes and improvements were necessary to continue further work on withdrawal and separation of starch during milling (Plate 5). A top bearing and new shaft were procured for the purpose of operating the mill at higher speeds. Spacing collars served to position the blades on the shaft, and also acted as reinforcement for the shaft while operating at higher speeds. A small fractional horsepower centrifugal pump (Eastern Engineering Co.) was used to deliver steep water from the steep tank to the mill feed hopper, which was connected to the bottom of the mill, at a maximum rate of 14 lb./min., or 1.8 gal./min. A variable speed drive connected to the screw conveyor in the mill feed hopper carried grits through the hopper to the downpipe at a controlled rate. A ½ H.P. bronze gear pump, connected to the top outlet of the mill, moved overflow grit slurry from the mill to the shaker, where milk and residual grits were separated. Since a continuous current of steep water was passed through the mill, the external circulating system previously mentioned was removed. The four sets of baffles supported on 3/8-inch vertical steel rods used in the foregoing runs were eliminated when it was found that liquid turbulence, induced by higher operating speeds, caused them to buckle inward toward the shaft, resulting in scoring of the shaft.

Under this modified arrangement, the runs were begun by setting the mill and screw conveyor in motion. The steeped grits were placed in the hopper, and steep water was run into the hopper at the rate of 1.8 gal./min., washing the grits into the mill. When the mill was filled to capacity, the overflow starch milk was pumped to the shaker, where any grits were separated and periodically returned to the mill feed hopper until the run was finished.

EXPLANATION OF PLATE V

Flowsheet of semi-continuous and periodic flushing starch recovery procedure.

PLATE V


MILL
FEED
HOPPER
MILL
STEEP
WATER
3
4
GRIT
SLURRY
GRITS
SHAKER
5
STEEP
TANK
1
STARCH
MILK
TANK
2
6
TO STARCH
TABLES
1 & 2 1/4 H.P. LIGHTNIN
MIXERS
3 FRACTIONAL H.P.
CENTRIFUGAL PUMP
4,5 & 6 1/2 H.P. GEAR PUMPS

Interrupted Batch Milling

In an effort to simplify the experimental work by eliminating the tabling operation and to confirm the effect of prolonged milling, a further series of runs was carried out by filtering the starch milk through canvas twill cloth under a vacuum of approximately 25 inches. Here the starch and gluten were not separated. For this series, the screw convey in the feed hopper was removed, and the steeped grits were flushed into the mill without any conveying motion. When large weights of grits were encountered, they were loaded through the top of the mill. Straight batch runs were made where the mill contents were left unchanged, as well as interrupted runs where the separated grits were re-loaded into the mill with more steep water for continued milling.

This interrupted milling procedure was investigated to determine definitely if an improvement in final yields could be obtained over the previous method of Banowetz (1) using straight batch milling. His results indicated that batches of like initial composition milled for increasing periods of time showed an increase in starch yield with time until a maximum yield value was reached. Further milling then caused the yield of starch to drop.

For this set of tests, the shaker was not used to separate the grits from the starch milk. Instead, the 40-mesh screen bucket was placed under the mill outlet after each milling interval, and the slurry was emptied through it. The mill was then rinsed with approximately two gallons of water, and this rinse was also run into the screen bucket. Where larger weights of grits were being milled, it was necessary to rinse additionally the separated grits free of adhering starch and gluten.

After complete rinsing of the grits, the starch milk was prepared for filtration by adding filter cel. The filter cel, a partially dehydrated silica gel, served as a filtration aid by helping to form a more porous filter cake and overcame, to some degree, the tendency of the milk to clog the filter cloth. The filter cel was added to the milk in the proportion of one ounce for every pound of raw grits milled.

After a cake was formed on the filter cloth, it was washed by stirring in two gallons of water and filtering. The washing was repeated, and the cake was scraped from the filter and dried under standard conditions.

Periodic Flushing

A final series of runs were made by periodically flushing steep water through the mill at the maximum rate of 1.8 gal./min. The overflow starch milk, containing some grits, was pumped to the shaker, and the separated grits were returned to the mill for further processing at the completion of the flushing period. A milling speed of 4,700 r.p.m. was used for most of these runs. Processing time varied from 15 to 30 minutes, with total flushing time never exceeding five minutes. When the run was completed, the mill was drained, and the mill and overflow lines rinsed with water. Then the grits were separated on the shaker, and the starch water was tabled.

Solids Content

For the purpose of making reproducible runs, it was necessary to determine the solids content of sorghum grits in various stages of processing. Hereafter, grits, or raw grits, will refer to the material as received. Dry grits are grits on a moisture-free basis, while steeped grits are grits that have undergone the steeping operation and have been allowed to drain at least 15 minutes.

Moisture determinations were carried out in an oven, under approximately 27 inches of vacuum, at 212° F. Batch 1 of raw grits, received December 6, 1949, had a solids content of 90.8 percent, while Batch 2, received April 23, 1950, contained 90.3 percent solids. Using these values, the solids content of steeped, drained grits was calculated from weighings made during 20 experimental runs. The average solids content of steeped, drained grits was 62.2 percent.

Solids contents were also determined on starches before and after drying. It was found, from three determinations, that solids contents of starches before drying ranged from 88.9 percent downward, depending on the amount of time elapsed between tabling and collecting of the starch. Solids contents of starch after eight hours in the tray drier were found from two tests to be 92.5 and 93.0 percent.

Wet grits were weighed, previous to drying, at the end of assorted runs, and their solids content found from their weight after drying. The first three entries in Table 2 indicate wet grit batches from which only previously separated water was drained. The last three determinations involved procedures which tended to increase the solids content.

Table 2. Solids content of wet, milled grits.

Run no.	Solids content percent	Remarks
A-19	27.8)	
A-22	21.3)	Average - 25.3 percent
A-28	26.9)	
A-14	35.7	Drained for short time on screen
A-13	40.2	Drained overnight on screen
A-36	43.7	Squeezed to remove water

Starch Product

A chemical analysis and an empirical viscosity test were used to evaluate starch quality and purity.

The empirical viscosity determination was carried out by a rotating cylinder viscometer, viscosity being measured at regular intervals, while the starch sample, suspended in water, underwent a specified heating and cooking period. This test was devised by Dr. H. N. Barham et al. (3), and determinations were made under his supervision at Kansas State College.

A 10 percent starch suspension was put in the rotating cylinder viscometer, operating at 60 r.p.m. A temperature of 20° C.

was established, and heating was begun at the rate of 0.5° C. per minute. Heating was continued through gelatinization stage until a temperature of 93° C. was reached. Temperature was maintained at 93° C. for 30 minutes, constituting the cooking period. The temperature was then lowered 0.5° C. per minute until a peak viscosity measurement was passed somewhere between 22° and 30° C. The torque, in grams, imposed on a freely suspended cylinder in the mixture, was measured throughout the cycle in order to obtain a smooth plot. Two viscosity peaks are obtained, one at a high temperature and the other during the cooking period. The ratio of the cold peak to the hot gives an index useful in determining the pasting qualities of the starch tested. Approximately three hours after completion of the viscosity test, the starch gel strength was found by noting the weight of steel shot required to move a special bucket, suspended on a pulley, through the starch gel.

DATA AND RESULTS

The letter before each run number indicates the mill which was used. Mill A was used for the majority of runs in this investigation.

Trial Runs

Runs LB-54 to A-6 constituted preliminary trials on the procedure of removing starch milk during the milling operation.

Table 3. Results of trial runs, 2,600 r.p.m.

Run no.	Milling time, minutes	Initial dry[1] grit conc. percent	Yield[2] Percent starch	Yield[2] Percent grits	Remarks
LB-54	15	11.6	48.2	15.0	Starch milk removal at 5-minute intervals
A-1	15	28.7	24.4	46.2	Continual withdrawal of starch milk, 1.4 gal./min.
A-2	30	28.7	39.4	16.3	Reverse grit recirculation begun. Starch milk removed between 5 and 10 minutes
A-3	20	7.1	53.8	25.6	Straight batch run
A-4	30	28.7	43.6	21.4	Removal of starch milk started after 10 minutes milling
A-5	10	28.7	36.9	30.0	Continuous separation of starch milk
A-6	20	28.7	41.8	26.2	Continual removal of starch milk

[1] Weight percent dry grits based on total slurry weight.

[2] Weight percent of starch and grits, as taken from the drier, on the basis of raw grit weight.

In Run LB-54, where 10 pounds of raw grits were milled at five-minute intervals (starch milk separated and replaced with fresh steep water every five minutes), a decreasing power requirement was noted as the milling progressed.

Table 4. Total power requirement for Run LB-54.

Milling intervals, minutes	Total power requirement, K.W.H. per minute
0 - 5	0.0099
5 - 10	.0075
10 - 15	.0071

A material balance was carried out on LB-54 by determining the solids content of the tabling effluent and considering the solids content of the raw grits and starch. The balance was met to an accuracy of 0.3 percent. The solids content of the steep water was considered negligible when compared to the accuracy in determining the solids content of the tabling effluent.

Table 5. Material balance on Run LB-54.

Component	Weight, pounds		
Raw grits[1]	10		
Dry grits		9.08	
Initial total		9.08	
Starch (from drier)[2]	4.56		
Starch (dry basis)		4.22	
Remaining grits		1.50	
Solids in tabling effluent		3.33	
Final total		9.05	
Loss		0.03	(0.3%)

[1] Solids content of raw grits 90.8 percent.
[2] Solids content of starch from drier taken as 92.8 percent.
[3] Solids content of tabling effluent 0.91 percent. Weight of tabling effluent, 366 pounds.

In this series of runs, the starch yield averaged three to five percent greater than comparable runs of Banowetz. This is attributed to two factors: The reversal of the direction of circulation, and the removal of starch milk during the runs.

Run LB-54 showed a reduction in percent grits remaining when compared to a straight batch run from the data of Banowetz (1).

Semi-Continuous Milling

Runs A-7 to A-10 were carried out by continual removal of starch milk from the mill, using steep water at 120° F. as a flushing agent. A conveyor screw in the hopper served to feed grits into the mill, with the aid of entering steep water. Overflow starch milk was pumped to the shaker where grits were separated.

It was noted that when low initial dry grit concentrations were used, no ground grits were carried over with the starch milk. As dry grit concentration was increased beyond 10 percent grit carryover took place, indicating tendency of the grits to settle to the bottom of the mill, in spite of the turbulence induced by the rotating blades.

In these runs, steep water was run through the mill in an upward direction at a rate of approximately one gal./min. The average capacity of the mill at these speeds is 3.4 gallons, indicating that the steep water took 3.4 minutes to pass through the mill. From Runs A-7 to A-8, control of tabling flow rate

Table 6. Semi-continuous milling.

Run no.	Milling time, minutes	Initial dry[1] grit conc. percent	Operating speed, r.p.m.	Yield[2] Percent starch	Percent grits	Remarks
A-7	30	30	3,200	-	18.8	Twelve minutes required to load mill fully
A-8	30	30	2,600	-	24.4	Nine minutes required to load mill fully
A-9	30	30	2,600	36.3	23.1	All grits fed to mill before milling started

1 Weight percent dry grits based on total slurry weight.
2 Weight percent of starch and grits, as taken from the drier, on the basis of raw grit weight.

had not yet been established, and the starch yield was not determined.

Run A-10 involved 20 pounds of raw grits and a milling time of one hour. At the start of the run, steeped, drained grits were fed to the mill through the feed conveyor at a rate of 0.5 pounds dry grits/min. A steep water flow rate of approximately one gal./min. was maintained. After 23 minutes' milling time, return of grits separated on the shaker was begun. Screened grits were returned for further milling at approximately four-minute intervals. Twenty-five percent of screened grits collected were discarded and replaced by an equal weight of steeped grits, not previously exposed to milling.

Flushing with hot steep water at 120° F. was continued until 44 minutes had elapsed, at which time all the steeped grits had been fed to the mill. Grinding was continued, but steep water flow was only used periodically to flush a small amount of screened grits, carried through the overflow, back into the mill. After 16 additional minutes of milling in this manner, the run was concluded.

Table 7. Yields from Run A-10.

Component	Yield percent
Starch	36.3
Grits	23.1

This procedure did not produce high starch yields, and the extent of grinding of the grits was of the same magnitude as for straight batch runs.

Interrupted Batch Milling

Runs A-11 to A-19, all of 15 minutes duration, were carried out to confirm the effect of prolonged milling on the sorghum grits. Starch was separated by filtering the milk under vacuum through a canvas cloth in the presence of filter cel. This residue could be more properly called a "starch fraction", since it contained a relatively large quantity of gluten.

For some runs of this series, decantation was used to separate liquid from the settled starch. This method was abandoned when it was found too lengthy, and the filtration procedure was substituted.

Some residual grits were separated from the starch milk and thorougly washed in the 40-mesh screen bucket. Very finely ground grits were removed in this separation step. For these reasons, the weights of the starch fraction had little significance and were not recorded. The efficiency of grinding was determined by the residual grit yield.

For this series of runs, a set of four blades similar in size to those previously mounted was placed at the bottom of the shaft, close to the mill floor, to overcome the tendency of the grits to settle during milling.

The effect of periodic removal of starch milk during milling was studied on a comparative basis between similar runs.

Table 8. Effect of interrupted batch milling[1].

Run no.	Number and duration of passes through the mill	Initial dry[2] grit conc. percent	Operating speed, r.p.m.	Percent grits[3]	Remarks
A-11	3 - 5 min. ea.	20.5	2,600	25.0	Starch by decantation
A-12	1	20	2,600	45.0	Starch by decantation
A-13	1	23.2	2,600	41.4	Filter-cel started
A-14	3 - 5 min. ea.	9.5	2,600	37.5	
A-15	1	31.9	3,200	21.9	
A-16	1	32.4	3,600	26.9	
A-17	3 - 5 min. ea.	31.8	3,600	20.6	
A-18	3 - 5 min. ea.	34.8	4,700	14.4	Low initial speed
A-19	1	35	4,700	21.9	Low initial speed

1 All runs of 15 minutes duration.
2 Weight percent dry grits based on total slurry weight.
3 Weight percent of grits, as taken from the drier, on the basis of raw grit weight.

It can be seen from Table 8 that a greater milling of grits took place upon removal of the starch milk and replacement with fresh steep water. The initial dry grit concentration of 9.5 percent in A-14 resulted in a higher grit yield than found for A-11, indicating that higher dry grit concentrations tended to cause a lower grit yield and, thus increased grinding efficiency.

The low initial milling speeds for A-18 and A-19 were caused by belt slippage around the shaft pulley. This resulted from the increased load of higher dry grit concentrations on the bottom blades recently installed. It was subsequently found that these bottom blades threw grits up into the feed pipe with enough force to cause occasional clogging when dry grit concentration exceeded 30 percent.

In Fig. 1 are plotted all straight batch runs (A-12, 13, 15, 16, 19) from Table 8 as operating speed against percent yield of grits remaining. Fig. 1 also shows a curve of operating speed against percent yield of grits for all runs where three passes of five minutes each were made through the mill (A-11, 14, 17, 18). As previously described, starch milk was removed from the grits after each five minute milling interval, and the partially ground grits were returned for another five minute milling interval in the presence of fresh steep water. Interrupted runs were made at the same dry grit concentrations as the corresponding straight batch runs. The curve for the interrupted batch runs lies consistently below that for the continuous batch runs, indicating that increased milling efficiency was brought

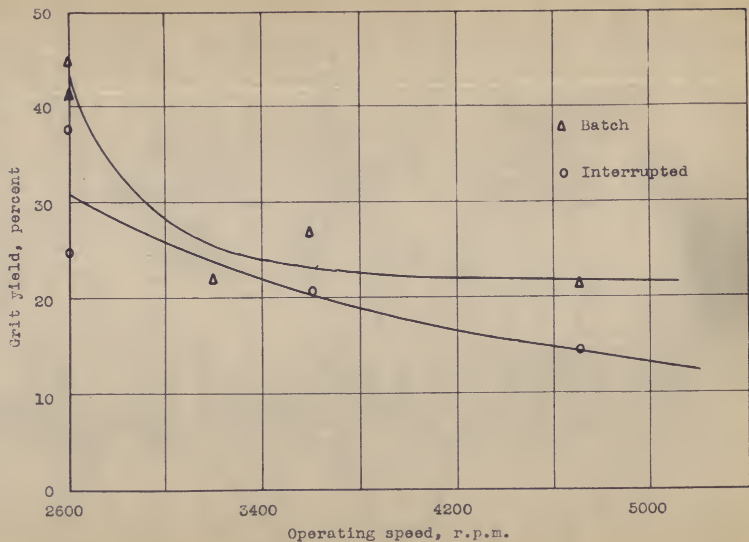


Fig. 1. Operating speed vs. grit yield.

about by the periodic removal of starch milk from the partially ground grits.

For Runs A-18 and 19, it was found difficult to separate the starch milk from the grits using the screen bucket, because the greater quantity of starch present settled out rapidly and formed an impermeable layer on the 40-mesh screen. This would indicate that an upper limit of initial grit concentration for the easy separation of starch milk was being approached.

Periodic Flushing

Runs A-20 to B-2 were made, for the most part, using the periodic flushing procedure. An effort was made to return all partially ground grits, carried over with the starch milk, back into the mill as rapidly as possible after the end of the flushing period. The return of overflow grits to the mill was accomplished within two minutes after the flushing period, depending on the weight collected on the shaker screen. More grits were washed over during the first flush than the following ones, since a greater quantity of grits were present in the early stages of milling. Twelve pounds of raw grits was the largest quantity used, and, under these conditions, approximately four pounds of partially ground wet grits were collected on the shaker screen during an initial flush of three minutes' duration. This weight of wet grits represents approximately one pound of dry grits.

The objective of Runs A-20 to B-2 was to find the conditions under which a maximum yield of starch could be obtained.

Table 9. Periodic removal of starch milk.

Run no.	Milling time, minutes	No. of flushes and time of occurrence	Initial dry grit conc. percent[1]	Operating speed, r.p.m.	Yield[2] Percent starch	Yield[2] Percent grits	Remarks
A-20	15	2 - 4- 6 min. 9-11 min.	25	4,700	-	14.4	
A-21	15	2 - 4- 6 min. 9-11 min.	19.7	4,700	-	15.1	
A-22	15	2 - 4- 6 min. 9-11 min.	16.1	4,700	-	18.9	
A-23	15	2 - 5- 7 min. 9-11 min.	25	4,700	51.0	17.3	
A-24	15	2 - 4½- 5½ min. 9½-10½ min.	21.2	4,700	46.6	21.9	
A-25	15	2 - 4½- 5½ min. 9½-10½ min.	10	4,700	51.3	24.3	
A-26	15	Straight batch run	26.6	4,700	44.2	22.1	Speed 4,000 to 4,400 rpm. in first 7 minutes
A-27	15	3 - 2½- 3½ min. 6½- 7½ min. 9½-10½ min.	26.0	4,700	50.5	19.4	
A-28	15	2 - 2½-3½ min. 5½-6½ min.	26.8	4,700	46.6	24.2	Bottom blade set removed for this and future runs

Table 9. (cont.)

Run no.	Milling time, minutes	No. of flushes and time of occurrence	Initial dry[1] grit conc. percent	Operating speed, r.p.m.	Yield[2] Percent starch	Percent grits	Remarks
A-29	20	2 - 5 - 6 min. $9\frac{1}{2}$-$10\frac{1}{2}$ min.	30.4	4,700	46.7	20.8	
A-30	30	3 - 5- 6 min. 9-10 min. 15-16 min.	36.6	4,700	51.0	19.5	
A-31	20	3 - $2\frac{1}{2}$- $3\frac{1}{2}$ min. $5\frac{1}{2}$- $6\frac{1}{2}$ min. $9\frac{1}{2}$-$10\frac{1}{2}$ min.	39.8	4,700	46.3	19.8	Difficulty in screening milk through shaker
A-32	25	3 - 1- 4 min. 7- 8 min. 13-14 min.	39	4,700	50.0	18.75	
A-33	25	3 - 1- 4 min. 7- 8 min. 13-14 min.	38.1	4,700	43.0	22.4	Milk from first minute flushing lost
A-34	25	3 - 1- 4 min. 7- 8 min. 13-14 min.	39	4,700	51.5	17.2	
A-35	25	3 - 1- 4 min. 7- 8 min. 13-14 min.	39	4,600	52.7	15.6	Remaining steep water tabled. Water spray on shaker used

Table 9. (concl.)

Run no.	Milling time, minutes	No. of flushes and time of occurrence	Initial dry grit conc. percent[1]	Operating speed, r.p.m.	Yield[2]		Remarks
					Percent starch	Percent grits	
A-36	25	3 - 1- 4 min. 7- 8 min. 13-14 min.	36.4	2,800	44.3	28.1	Milling carried out at 100° F. Low speed due to belt slippage
B-1	25	3 - 1- 4 min. 7- 8 min. 13-14 min.	18.9	3,600	43.7	31.3	Cloverleaf shell. Milling carried out at 110° F.
B-2	25	Straight batch run	17.3	3,600	37.0	33.3	Milling carried out at 110° F.

[1] Weight percent dry grits based on total slurry weight.

[2] Weight percent of starch and grits, as taken from the drier, on the basis of raw grit weight.

For dry grit concentrations exceeding 25 percent, it was found that the bottom blade set threw the grits up into the feed pipe and caused occasional clogging. For A-28 and subsequent runs, this blade set was removed so that higher initial dry grit concentrations could be milled. For this reason, Runs A-20 to A-27 will be considered separately from the remaining tests made.

Using the bottom blades, it was found that up to an initial dry grit concentration of 25 percent, increasing dry grit concentration resulted in greater milling of the grits. Fig. 2 shows a plot of dry grit concentration at the start of Runs A-20, 21 and 22 against the final percent yield of grits. Milling conditions were alike for these three runs.

A greater total flushing time tended to cause increased milling of grits and greater starch yield. Runs A-25 and 27, made using periodic flushing, gave higher starch yields and lower grit residues than A-26, which was a straight batch run. However, since the milling speed was low during the early part of the batch run, the results are inconclusive.

The starch yield could not be predicted from the yield of grits obtained. The two quantities appeared to vary independently.

After removal of the set of bottom blades, higher initial dry grit concentrations were milled. From the standpoint of industrial economy, it is desired to use as high a grit concentration in the mill as possible, since the amount of tabling effluent to be handled, either as recycle water after the removal of gluten, or as waste, would be reduced.

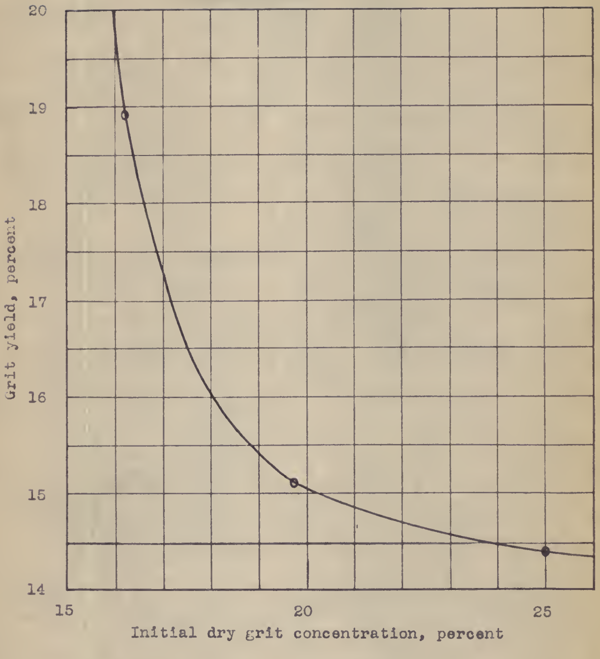


Fig. 2. Initial dry grit concentration vs. grit yield.

In the series of Runs A-28 to B-2, it was found that initial dry grit concentrations exceeding 40 percent resulted in inefficient separation of starch milk from grits carried over during flushing. The greater amount of starch present adhered strongly to the grits and was difficult to separate. This separation of starch on the shaker was improved to some degree by the use of the fine spray of water directed on the overflow slurry as it moved down the 200-mesh screen.

With an initial dry grit concentration of approximately 40 percent, a maximum starch yield of 52 percent was obtained by milling for 25 minutes with flushing periods occurring at periods of one to four minutes, seven to eight minutes and 13 to 14 minutes during the milling interval; or a total flushing time of five minutes. The percent of grit residue at the end of this procedure ranged from 15 to 22. Thus, approximately 25 percent of the original solids were present in the tabling effluent.

In Run A-33, the milk obtained from the first minute of flushing was lost, causing a drop in starch yield of approximately seven percent, total basis. Banowetz (1) has shown that the greatest milling of the grits takes place within the first five minutes, and it would be expected that the loss in starch yield would be greater than the value determined. It is possible that insufficient separation of starch from the overflow slurry during the first minute of flushing could contribute to this difference.

In Run A-35, starch contained in the steep water remaining at the end of the run, approximately seven gallons in volume, was separated on the tables after the main portion of milk had been tabled. The total starch collected was then dried and weighed, giving a starch yield of 52.7 percent. This yield was one to two percent greater than obtained without processing of the remainder of steep water.

In Run A-36, tests were begun to determine the effect of lower milling temperatures on the starch yield.

In Runs B-1 to 2, a new shell having a cloverleaf shape was used. This modified shape was adopted in an effort to make the grit contact the blades more frequently than would take place in a circular shell.

Both batches were milled at 110° F. A greater starch yield was obtained by periodic flushing out of the starch milk than by straight batch operation.

Starch Quality

Sample A-34 was submitted to Dr. H. N. Barham's laboratory, Department of Chemistry, Kansas State College, for evaluation. The viscosity test described under heading II, Materials and Methods, Barham et al. (3), was performed on the sample. The test indicated that the sample was satisfactory and had good pasting properties, although it contained some protein and also foreign matter. The foreign matter was most probably introduced from the starch milk tabling lines.

Fig. 3 is a graphical record of the viscosity test performed on sample A-34. It is characterized by a sharp peak at the hot

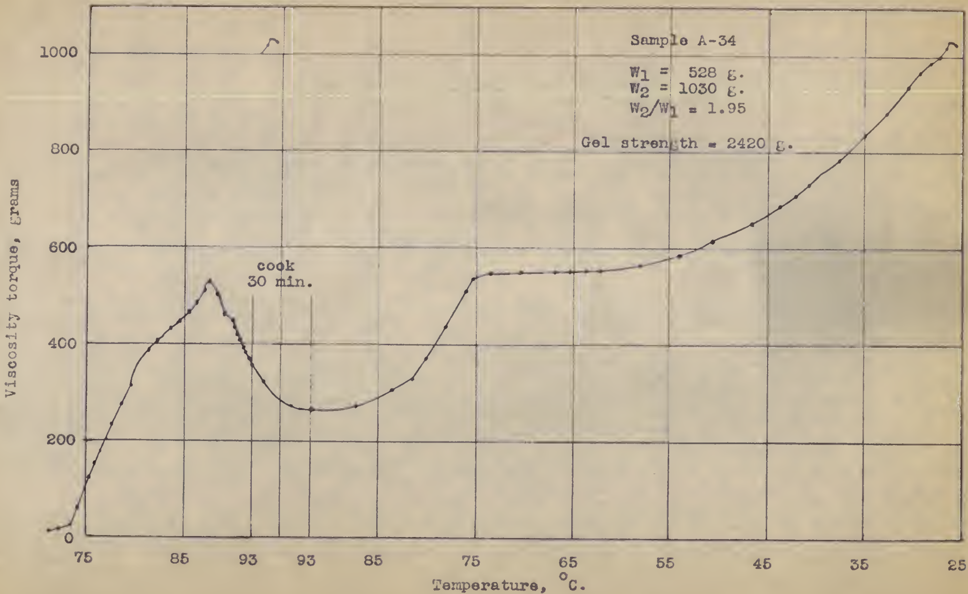


Fig. 3. Starch viscosity vs. temperature.

viscosity maximum (marked A). Barham (2) states that this may possibly be caused by a selective starch recovery process wherein one particular starch fraction is recovered more favorably than others.

Power Consumption

The rate of energy consumption during milling was determined in selected runs at various intervals of time in K.W.H./min.

The power requirement decreased with increasing milling time, except when shaft bearings became worn. In such cases, the power would rise with milling time and then fluctuate. The fall in power requirement with increased milling time would indicate that the power required for milling was proportional to the dry grit concentration at any given time.

The "no load" requirement, which is the rate of energy consumed when the mill operates with a full capacity of water only, varied from 0.0273 to 0.0360 K.W.H./min. at 4,700 r.p.m. The arithmetic average of six determinations gave a value of 0.0316 K.W.H./min.

Data from Run A-26 gave a representative picture of the power requirement during the milling operation. The total milling time was 15 minutes, and the run was of straight batch type.

Table 10. Power requirement during milling for A-26.

Time of milling, minutes	Total power requirement, K.W.H./min.
5	0.0418
9	0.0381
11	0.0381
14	0.0322

The data from Table 10 are plotted on Fig. 4.

The area under this curve was integrated graphically to give a total energy consumed in grinding of 0.5901 K.W.H. From this was subtracted the average no load power consumption, giving an approximate figure of 0.1161 K.W.H. for the energy consumed in the actual milling of the sorghum grits. This energy, on the basis of 15 minutes, is equivalent to .623 H.P. Assuming that all gluten was removed from the grits at the end of the milling time, approximately four pounds of starch was present in the milk at the termination of the run. The energy requirement of this run could then be expressed as 0.029 K.W.H. per pound of starch produced.

DISCUSSION

While experimental runs were being conducted, effort was made to improve the methods used and to eliminate all variables in the process except those under study. These refinements, among others, included:

a. The consideration of moisture contents of raw material and final products in calculating percent yields.

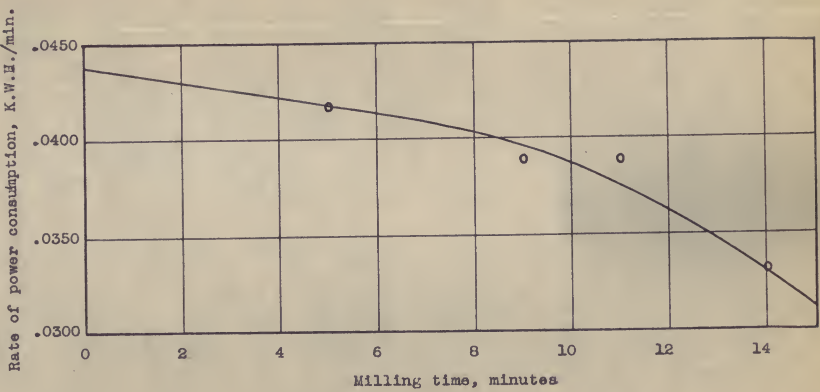


Fig. 4. Rate of power consumption vs. milling time.

b. Setting up of the piping carrying starch milk to the tables so that the flow rate to the starch tables was constant and could be varied when desired.

c. Specifying a standard drying procedure for final starch and grit yields to give reproducible results.

d. Various mechanical adjustments of a secondary nature.

It was seen, from the foregoing data and results, that increased milling of sorghum grits and a greater yield of tabled starch resulted when previously recovered starch, in the form of starch milk, was removed from the mill while milling of the residual grits was continued. Prolonged exposure of previously separated starch to prolonged milling, as encountered in straight batch operation, lowered the yield of tabled starch and decreased the degree of milling of the sorghum grits.

However, examination of Table 1 shows that the quantity of starch recovered by the hydraulic milling process was still far less than that available in the raw sorghum grits. The typical analysis of Westland Milo sorghum grits (2) on a dry basis shows the starch content to be approximately 83 percent. The highest starch yield obtained by the hydraulic milling method was 53 percent, while the corresponding residual grit yield amounted to 16 percent (A-35, Table 9). These figures were found to be limit values for the milling of slurries at an initial grit concentration of 40 percent, using a periodic flushing procedure. Referring to the residual grit yield of 16 percent, it is evident that more starch is removed from the sorghum grits than is finally collected from the tables. Assuming the most unfavorable condition, that all gluten and extraneous carbohydrates have been removed from the residual grits, a grit yield of 16 percent should produce a starch yield of approximately 69 percent, which is higher than actually attained. On this basis, the present milling procedure removes 83 percent of starch available from the raw sorghum grits.

No unaccounted-for losses were noted in these experimental runs, except possibly for small quantities of residual grits which could not be completely scraped from the shaker screen for final collection. The material balance on LB-54 showed no major material losses occurring in the starch recovery process (Table 5).

While the periodic flushing process increased starch yields to some extent, it may be possible that for the initial dry grit concentrations and operating speeds used, the flushing periods may have been too short and/or the flushing flow rate too low to achieve the maximum starch yield. The difficulty in separating recovered starch from overflow grits encountered in some runs would tend to defeat the purpose of periodic flushing.

For the flushing procedure finally established in Runs A-32 to B-2, the total amount of steep water flushed through the mill (at 1.8 gal./min.) was nine gallons, or 74.2 pounds. The operating capacity of the mill at the operating speed of 4,700 r.p.m. was approximately 2.4 gallons, and the fluid capacity of the mill (total volume minus volume occupied by partially ground grits) had an average value of 1.67 gallons. An arithmetic average was used to approximate the fluid capacity, since the grit volume steadily decreased during the milling interval. A figure of 16 lb./gal. for the density of freshly steeped grits was used. On this basis, the fluid contents of the mill were changed approximately 5.4 times during the run--a greater flushing than had been attained in previous procedures.

Initial dry grit concentrations appeared to have little effect on starch yield in this work. However, the efficiency of the flushing operation could be increased by the use of lower initial dry grit concentrations, or by devising means of keeping the grits from being carried from the mill during the flushing period.

However, the use of a dilute grit slurry for milling would materially increase the quantity of liquid to be handled for each pound of starch produced in a batch milling process. The disposal of tabling effluent as a waste or the purification of effluent, by settling out of gluten, for use as recycle water, constitutes a major problem in the starch industry, because of the large volumes of liquid involved. It would seem, therefore, that the milling of a dilute slurry of grits could best be accomplished on a fully continuous basis. Such a process would involve the continual entry of freshly steeped grits and the continual removal of ground grits and starch milk. The concentration of starch in the continuously withdrawn starch milk would have to be great enough to make the processing of the tabling effluent economically feasible.

To increase the yield of starch obtained from sorghum grits by the hydraulic milling process, it is necessary to bring about greater milling of the grits than has previously been attained. This may be done by operating the hydraulic mill at higher speeds and by improving the overall design of the mill so that breakdown of the grit structure takes place more completely.

The tabling process should be investigated to confirm that the separation of starch from the starch milk is taking place at maximum efficiency. In this connection, Kerr (5) states that for cornstarch, peak separation of starch during tabling takes place when the starch milk is at a pH between 3.8 and 4.2. Further, separation of starch is more readily accomplished from a dilute slurry than a concentrated one. Tabling of cornstarch slurries is carried out at 12° Bé. Occasionally, the presence of small quantities of dissolved electrolyte, in addition to an acid, facilitate starch separation. The effect of these three variables on the tabling efficiency of cornstarch are interdependent.

Though sorghum starch is being studied, the effect of these variables on its separation from starch milk should not be overlooked.

CONCLUSIONS

The hydraulic milling of sorghum grits is accomplished to a greater degree by the periodic removal of starch milk and its replacement by steep water than by straight batch milling.

The starch yield obtained by the hydraulic milling of sorghum grits is greater when starch milk is periodically removed during milling than by straight batch milling.

A greater degree of milling, either by use of higher milling speeds or improved mill design, is needed to obtain the full yield of starch from the sorghum grits.

The efficiency of the tabling operation should be investigated to see that maximum separation of starch from starch milk is being attained.

The hydraulic milling of sorghum grits produces a starch of good pasting quality.

ACKNOWLEDGMENT

Appreciation is expressed to Professor William H. Honstead for his guidance and suggestions in conducting this investigation on the hydraulic milling of sorghum grits.

Through the helpfulness of Dr. H. N. Barham, Department of Chemistry, fundamental information on the properties of sorghum grits and sorghum starch was made available, and tests were conducted on the starch samples.

BIBLIOGRAPHY

(1) Banowetz, Leonard
Department of Chemical Engineering, Kansas State College. Unpublished data, 1949.

(2) Barham, Harold N.
Department of Chemistry, Kansas State College. Unpublished data.

(3) Barham, Harold N. et al.
Kansas State College Agricultural Experiment Station Bulletin 61. 47 p., March, 1946.

(4) Johnston, Rodney W.
M. Sc. thesis, Kansas State College. 1941.

(5) Kerr, Ralph W.
Chemistry and Industry of Starch, New York: Academic Press, 1944.