Wet Milling
of Cereal Grains

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Orlando Jones, an Englishman, was a pioneer whose work in 1840 founded a multimillion-dollar industry that touches all our daily lives. Every early starch producer used his process, in which an alkali was used to recover the starch. The process worked so well that the industry grew by leaps and bounds until, by 1880, this country's yearly consumption was estimated at 200 million pounds. Thus the cornstalk industry was born.

Wet milling, which has been considerably modified from Jones' original method, now consists of grinding the soaked grain and then separating the starch with water. Eleven companies were operating 14 wet-milling plants in 1950. They used about 6 percent of our corn crop, or 140 million bushels. Three of the plants have occasionally wet milled grain sorghums since about 1945. Another firm has announced plans for wet milling about 2 million bushels (56,000 tons) of grain sorghum annually. Wheat starch and rice starch have been made by various wet-milling procedures. Starch also has been obtained from barley and oats in the laboratory.

This use of the cereal grains has given the starch industries the advantage of a raw material high in starch, easy to store, and available in quantity. Every cereal grain can be wet milled by modification in equipment or processing. Because of the differences in the physical properties of starch from various sources, the starches may not be interchangeable for some purposes. Therefore, the production of starch from cereal or tuber crops depends upon the demands of industries for products most satisfactory for their purposes.

Cereal grains can be roughly classed as waxy and nonwaxy, or common. The terms do not imply the presence or absence of wax, but are merely descriptive of the physical appearance of the inside of the whole kernel. Waxy varieties are found in corn, grain sorghum, barley, rice, millet, and Job's tears. Commercially, only small amounts of the waxy corn and sorghum have been processed for starch.

Because sound grain yields the most starch with the least processing difficulty, wet mills as a rule process grain of grade No. 2 or 3. Only corn and sorghum are wet milled today.

The oldest wet-milling method for extracting wheat starch is the Halle or fermentation process, in which the grain, softened by steeping in water, is ground and made into a mash with water and allowed to ferment a week or two. The modified gluten can then be washed out of the starch. A second method, known as the Alsatian process, consists of steeping, grinding, and washing out the starch, without previous fermentation. The methods have been replaced by the Martin process and the batter process, both of which are wet-separation, not wet-milling, methods. Wheat starch was also made during the Second World War by a modification of the corn wet-milling process developed at the Northern Regional Research Laboratory.

Rice starch is obtained by steeping the grain for 24 hours in 0.3 percent caustic soda solution, then grinding it
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with additional caustic soda. The starch is obtained after screening, sedimentation, and centrifuging. Sulfur dioxide has also been used instead of the caustic soda.

The process as described for extracting starch from corn applies generally for grain sorghum.

Most starch factories start with shelled corn delivered in railroad boxcars, each holding 1,200 to 1,800 bushels. A sample is collected from each car and analyzed. The grain is unloaded by means of power shovels, air conveyors, or tilters. It is weighed and then may go to either storage bins or a cleaner to remove dust, chaff, cobs, stones, rodent excreta, insects, broken grain, and other foreign material. In some plants the cleanings, except the larger particles, are conveyed to the feed system and mixed to form part of the gluten feed.

The cleaned grain is stored in bins in an amount sufficient to fill a steep tank. Some of the larger wet-milling plants are equipped with grain driers to dry high-moisture grain. Corn with a moisture content under .16 percent can usually be stored safely during the winter, but corn that contains more than 16 percent moisture is turned in the bins periodically.

Steeping, or soaking, is the most important step in wet milling. It loosens the bond that holds the starch granules together in the kernel; it removes solubles, mainly mineral matter, from the germ so that it is lightened and can be floated off in the germ separators; and it softens the kernel for grinding. Steeping does not harm or modify the starch very much.

Sulfurous acid water is used for the purpose. Steeping is done in a series of tanks in which lactic acid fermentation is controlled by a countercurrent flow of steep water and the addition of sulfur dioxide. Each tank holds 2,000 to 3,500 bushels of grain.

The sulfurous acid is made by burning sulfur and absorbing the sulfur dioxide in gluten settler water. This acidic steep water, amounting to 7 to 14 gallons per bushel and containing 0.07 to 0.25 percent sulfur dioxide, is heated to 116° to 133° F. and put on the oldest corn in the steeping system. The acid water is transferred countercurrently through the steeps and at times is circulated over each steep. The oldest corn, therefore, is soaked or steeped with water containing the smallest amount of solubles and the newest corn with water containing the largest amount of solubles.

The corn is completely covered during the steeping. The steeping time varies from 28 to 48 hours. The temperature of the contents of each steep is maintained at 116° to 133° F. by means of tube and shell heat exchangers or by sparging (injecting) the steam directly into the water. The water drained off the newest corn is called light steep water. It amounts to 4 to 10 gallons to the bushel and varies greatly in its composition. Soluble or colloidal matter amounting to 1.5 to 9.5 percent of the dry substance of the original grain is removed in steeping. The steeped corn, when withdrawn, has a moisture content of 40 to 55 percent, depending upon the condition of the grain used. More sulfur dioxide is required for steeping moldy and heat-damaged corn.

The starch is separated from the steeped corn in four steps. In the first, the grain is ground by degerminating mills to free the germ, which is then floated off in large tanks known as germ separators. Then, the underflow from the separators is screened and the resultant hull and grits are ground by means of buhrstone mills or vertical hammer mills. The third step is screening to separate coarse and fine fiber from a starch and gluten slurry. The germ, coarse fiber, and fine fiber are washed free of starch and gluten by a countercurrent flow of water over three or four sets of screens. The starch is separated from the gluten in the fourth step by tabling or centrifuging.

The trend today is to replace the tables with centrifuges because there is
less chance of any contamination, processing time is shorter, less floor space is required, and results are better.

The starch from the tables or centrifuges is filtered and washed on the filters to remove soluble matter.

Starch from the filters is dried to 5 to 18 percent moisture by kiln, belt, rotary, vacuum, spray, or shelf driers. The starch that leaves the driers with 10 to 14 percent moisture is known as pearl starch. It can be packed in 140-pound jute, burlap, or cotton bags, or in 100-pound paper bags. This starch will have a protein content of 0.26 to 0.38 percent on a dry basis and contain 0.5 to 1.2 percent total impurities, which include protein, ash, solubles, and oil.

Pearl starch is pulverized to produce food and powdered starch. The main difference between them is that food starch must not contain more than 0.005 percent of sulfur dioxide. Pearl starch may be dried further either before or after it is pulverized. To avoid explosions the moisture content has to be kept above 7.5 percent while the starch is being pulverized. Therefore, to reduce the moisture below 7.5 percent the starch is dried after pulverizing. The powdered starch is sifted through 200-mesh screens and bagged. Pulverized starch contains 5 to 14 percent moisture, depending upon its intended use.

Byproducts are recovered by drying the wet material from the milling system in flash driers, rotary fire driers, or rotary steam-tube driers. Two passes are generally used to maintain a more uniform moisture content in the product. Flash driers are used to dry gluten feed, gluten meal, and zein. In other installations, rotary fire driers are generally used for the first pass and steam-tube driers for the second pass. The rotary driers are used to dry gluten feed, gluten meal, and germ.

The washed germ from the screens is pressed with a screw- or disk-type water extractor and then dried with two passes through rotary driers. The dry germ will contain 42 to 50 percent oil and 14 to 18 percent protein. The oil is recovered by screw-type oil presses or by a combination of screw presses and solvent extractors. The resulting cake or meal, which contains 0.5 to 15 percent oil, 19 to 22 percent protein, and 3 to 6 percent moisture, is called oil cake or corn-germ meal. It may be sold as such, but most of it is added to the gluten feed. The oil is shipped out as crude corn oil or is refined in the plant.

The gluten liquor or overflow from the tables or first-pass centrifugals is concentrated by centrifuges or settlers. Most of the gluten settler water or overflow from the settlers is used to make the steep acid. The rest is mixed with the other process waters and used to wash the germ and fiber.

The settled heavy gluten is mixed with some fine fiber and filtered with recessed-plate presses or a string-discharge drum filter. The filtrate is mixed with the other process waters to wash the germ and fiber. The filter cake is dried by two passes through rotary driers. The tailings from screening the dried material pass into a hammer mill and can be added to the gluten meal or gluten feed. The material through the screen is called gluten meal and will contain 40 to 45 percent protein and 8 to 14 percent moisture. About 75 percent of the gluten can go into gluten meal and the remaining 25 percent into gluten feed.

The light steep water is evaporated from 2° to 5.5° Baume (average about 4° Baume) to 19° to 26° Baume. Small plants commonly use single-effect and larger plants triple-effect evaporators. The heavy steep water, which contains 35 to 48 percent dry substance protein and 2 to 16 percent dry reducing substances, is loaded into barrels or tank cars or is mixed in the gluten-feed system.

The coarse fiber from the screens is dewatered with screw- or disk-type water expressers. The excess, about one-third of the fine fiber not used to make gluten meal, is used in making
gluten feed. In some plants all the gluten and fine fiber go into a gluten feed of 27 to 29 percent protein. The chaff and screenings from the grain elevator may be added to the dewatered coarse fiber; then heavy steep water is added, and the mixture is passed through driers. If all the gluten is used, it is best to dry it separately through one pass of driers. The dried mixture from both sets of driers can be soaked with more heavy steep water and dried to a final moisture content of 9 to 14 percent. The oversize from the gluten-meal system and refinery mud (cake from the dextrose and sirup filters containing about 30 percent oil and 15 percent protein) is added before the final drying. Tailings from the pulverized-starch and dextrin screens and off-grade batches of starches or dextrins are also added to gluten feed.

Sweet gluten feed is made by adding hydro, a byproduct molasses from dextrose manufacturing, to the gluten feed before the final drying.

Starch can be converted into a number of simpler substances. Almost any acid can be used to catalyze the reaction of starch with water to form them. Time of conversion, temperature, and acid concentration are factors that can be varied to produce different conversions. Enzymes also can be used to convert starch into sirups.

The greatest acid conversion yields dextrose; lesser conversion yields maltose and the dextrins, along with smaller amounts of dextrose. The degree of conversion is expressed as dextrose equivalent (D. E.), which is a determination of the total reducing substances as dextrose. Products with a lower dextrose equivalent, that is, D. E. 30 to 70, are glucose sirups; those of a higher dextrose equivalent, D. E. 70 to 90, are dextrose sirups from which dextrose sugars are isolated by crystallization.

In the past, this conversion to sirups or dextrose took 4 to 14 hours under atmospheric pressure in large wooden tanks. Today the conversion takes 5 to 30 minutes under pressure in bronze batch converters or in pipe continuous converters.

Starch that contains the smallest amount of impurities is preferred for conversion. The starch cake from the filters is suspended in water to attain a gravity of 10° to 26° Baumé. This slurry is mixed with acid to obtain a pH of 0.9 to 2.4, approximately 0.02 to 0.50 percent acid. The higher acid concentrations are used for dextrose sugar conversions; the lower ones for glucose sirup conversions. The mixture is pumped continuously through a steam-jet heater into a chamber attached to the hydrolysis coil. A valve at the end of the coil controls the flow of the material and thereby the pressure and temperature. A vacuum flash chamber at the discharge is used for a partial concentration of the liquor. The retention time in the continuous converter will vary from 15 to 30 minutes. The liquor is mixed with a diatomaceous filter aid and pumped through a precoated filter to remove insoluble protein and fats (refinery mud). A partial or complete (4.0 to 5.5 pH) neutralization may be employed before the filtration. Some of the proteins and fats may be recovered by using skim tanks ahead of the filters and after the neutralizer.

The clarified juice is run through a triple-pass ion exchanger to remove the impurities. The ion-exchange resins are regenerated with acids and alkalis. The liquor from the ion exchanger at a pH of 4.5 to 6.0 has residual color removed by activated carbon. This treatment consists of contacting the juice at a temperature of 178° to 190° F. with activated carbon for 1/2 to 2 hours. Boneblack (char), activated carbon, or resinous absorbents may be used. Sodium bisulfite is added to the juice to preserve it, to aid bleaching, and to maintain 0.002 to 0.005 percent sulfur dioxide in the finished sirup. The liquor is evaporated to 42° to 45° Baumé for
glucose (corn) sirup. The sirup is sold in tank cars, drums, cans, and bottles. It also may be spray dried.

The process of converting the starch to dextrose sugar starts the same way, except that more acid and a higher temperature or longer time are used. The deionized, bleached, and clarified liquor is evaporated to 38° to 40° Baume and pumped into crystallizers. Because dextrose can crystallize in different forms, the liquor in the crystallizer is seeded with dextrose crystals from previous batches. From 20 to 30 percent of the dextrose goes back as "seed." After 80 to 140 hours of gradual cooling, the mass from the crystallizer is transferred to centrifuges to separate the dextrose crystals from the mother liquor (hydrol). The crystals are dried in two passes through rotary driers using clean hot air. The dextrose sugar may be packed "as is," or it may be ground to pass a 200-mesh screen. Generally, only 100-pound paper bags are used because of the hygroscopic nature of product.

Crude corn sugars are made by cooling the heavy dextrose liquor in forms. In this case the hydrol is included along with the dextrose. The lumps are broken up and then packed.

The modified starches include those that have been modified to form products of varied chemical and physical characteristics. These can well be grouped as the acid-modified, alkali-modified or pH-adjusted, and special starches and starch blends. The acid-modified starches yield thin-boiling (thin-bodied) or low-viscosity starches; the alkali-modified or the pH-adjusted starches yield thick-boiling (thick-bodied) or high-viscosity starches. Various chemicals are used to form special starches, such as nitrated starch, allyl starch, solvent-extracted starch, cold-water-soluble starch, chlorine-treated starch, and aldehyde-treated starch. Starch or any of the modified starches may be blended together or with other substances, such as tapioca flour, sago flour, wheat flour, oils, sulfonated oils, soaps, borax, and various chemicals, to form starch blends. The blends are used mainly for adhesives.

The acid-modified starches form the bulk of modified-starch production. They are prepared by treating starch slurries in large wooden tanks with sulfuric acid or hydrochloric acid maintained at a temperature below the gelatinization point of the starch for 10 to 24 hours. Acid of 0.002 to 0.8 percent concentration in the slurry is used. When the desired fluidity (viscosity) is reached, the conversion is stopped by adding an alkali such as caustic soda or soda ash. The resultant modified starch is filtered and dried like regular pearl starch. Some of the acid-modified starches may be pulverized. They are packed in 140-pound jute, burlap, or cotton bags, or 100-pound paper bags.

The alkali-modified starches are prepared by treatment with alkaline chemicals, followed by filtering and drying in the ordinary manner, or by pumping the slurry directly on to a rotary drum drier.

Brewers' grits is a special starch which is partly gelatinized by heating a starch slurry just under a gelatinization point. Unless great care is taken, the starch in the tank will gel or filtering and drying the product will be difficult.

Another special starch, which is used as a core binder and an additive to drilling mud, is made by gelatinizing and drying table liquor on a rotary drum drier. It contains 4 to 7 percent protein.

Dextrins are starch degradation products yielded by the dry conversion of starch of 5 to 10 percent moisture content with various catalysts. As in all the other conversions, time of conversion, type and amount of catalyst, and temperature are governing factors, which are varied to obtain different dextrins. Heat-degraded starch products can be classified as white dextrins, yellow (canary) dextrins, British gums, and blends. The commercial dextrins are not true dextrins but a mixture of...
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dextrins. Most British gums are made without a catalyst.

Pulverized starch for dextrin conversion is stored in a bin. From there it is conveyed to an acidifier—a blender that is a horizontal oblong enclosed wooden tank with openings and a horizontal mixing agitator. Dilute hydrochloric acid or other chemical catalyst is sprayed in over the starch and mixed in. After thorough blending, the starch goes into dextrinizers, where it is held at a fixed temperature between 150° to 400° F, for 1 to 18 hours. Indirect heating with steam or Dowtherm is preferred owing to the fire and explosion hazard of this operation. A totally enclosed jacketed steel kettle is used. It is also very important to avoid undue local overheating. The batch is dropped from the dextrinizer when it approaches the color and fluidity of a standard. It is then cooled, screened, blended with other like batches, and packed in 140-pound jute, burlap, or cotton bags, or 100-pound paper bags.

A certain dextrin may be blended with any of the starch products, that is, starch, modified starch, another dextrin, or British gum, or with chemicals to obtain blends with special properties. These blends are generally used for adhesives.

Adhesives have such varied uses that it is difficult to describe manufacturing procedures without mentioning each particular adhesive. They can be classified, according to their physical state, as liquid, paste, and dry. Dry adhesives may be modified-starch blends, dextrin blends with other materials, or drum-dried products. Dry adhesives can be further classified as to their solubility in hot or cold water.

Various chemicals are used to impart certain properties to an adhesive. Borax is used in almost every one to increase the viscosity and tack. To obtain a still greater viscosity and tack, alkalies, such as caustic soda or soda ash, are added. Urea, iodides, thiocyanates, chloral, and other chemicals are added to increase the cold-water solubility of an adhesive. Wetting agents like sulfonated castor oil are used in adhesives as defoamers and to wet difficult surfaces and thereby increase the bond. Hygroscopic materials, such as glycerol, ethylene glycol, sorbitol, glucose, calcium chloride, ammonium nitrate, urea, and sodium acetate, are used to keep the adhesive film from drying out and becoming brittle. Preservatives are required for almost every liquid or paste adhesive. Among the compounds used for this are sodium salt of o-phenylphenol (Dowicide A), sodium salt of pentachlorophenol (Dowicide G), formaldehyde, formalin, copper sulfate, zinc sulfate, fluorides, benzoates, sodium bisulfite, and mercury compounds. To impart more body and lessen the tendency for the liquid adhesive to set back, flours from wheat, rye, or sago may be added.

In general, the liquid and paste adhesives are made in small batch tanks. The various ingredients are added to hot water and held at a certain temperature until the desired product is obtained. A number of small batches are blended to yield a more uniform liquid adhesive, which is then barred.

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The preparation of starch from cereal grains was described by Marcus Porcius Cato in 184 B. C. Pliny the Elder attributed the first extraction of starch from grain to the inhabitants of the Isle of Chios. Until the latter part of the eighteenth century, starch was practically always obtained from wheat.