EFFECT OF DRYING TEMPERATURE ON SOLUBILITY OF CORN PROTEIN

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INTRODUCTION

After grains mature, they need one to three weeks in the field before they are dry enough to store. The longer the grain is left to dry in the field after it matures, the larger will be the field losses from lodging, drooping of ears of corn, and shattering. In some areas where insects are a serious problem, a late harvest increases the hazard of insect damage, both in the field and in storage. Being able to harvest as soon as the crop matures, rather than waiting for field drying, is of special significance to the corn farmer, because much of the corn grown in the Corn Belt is harvested by use of the picker-sheller or the corn combine which operate with least loss when the grain is at high moisture content. 20 to 30%. In addition, there is often the need to harvest when the equipment is available and before fall rains make the ground too wet to support heavy machinery. Finally, the grower often wants to sell the grain early, when the market price is high.

Since shelled corn packs more closely and allows less movement of air through its bulk in bins than ear corn in cribs, it dries more slowly in storage. Shelled corn at high moisture content must be artificially dried for safe storage, or it will mold and heat.

Adams et al. (1) and Hathaway et al. (2) reported that high temperatures used in artificial drying of corn would cause physical and chemical changes in the corn kernel that

would decrease the value of the grain to dry-millers, wetmillers, cattle feeders and distillers. The decrease may
result from lower yields of the desired product and/or from
difficulties in plant operation and production of low quality
products, as well as from decrease in nutritional value.
Behavior in the wet-milling process of corn dried at high
temperatures indicates that heat-treatment changes the
physical nature of the protein.

In the present study, the solubility of protein fractions obtained from corn dried at different temperatures was determined in several solvents.

REVIEW OF LITERATURE

Artificial drying with heated air

The amount of moisture contained in grain has a definite effect on its suitability for harvesting, handling and processes such as storage, feeding, germination, and milling of various kinds. For many processes there is an optimum or critical moisture content above or below which the results are not satisfactory. While grain is growing, the moisture content is high. As it ripens the percentage of moisture decreases, and moisture normally continues to leave the kernels after ripening. It would not be desirable to dry the grain completely even if it were convenient and economical to do so. Perhaps the most important practical reason for concern about the moisture content of grain is that molds, yeasts and other microorganisms require moisture for their growth. They do not obtain the necessary moisture unless the grain contains above 12% moisture and many molds will not germinate and grow in grain of less than 13.5%. Insects grow best in grain at 12 to 14% moisture content. The exact level at which the grain will be infested by insects and microorganisms depends upon the kind of grain, the temperature and the kind or species of organism.

Grain can be badly damaged by improper drying; overheating during drying is especially dangerous. Types of damage that may occur to grain as a result of overheating include: loss of germinability, scorching, hardening of kernels which leads to milling difficulty, reduction in baking quality in the case of wheat, checking which results in broken kernels and reduction in palatability or nutritive value in feed grains. Molding during drying may occur if the drying process takes too long.

Temperature is not the only criterion for "safe" heating. Drying difficulties have been due primarily to lack of understanding of technical relationships of drying to temperature, relative humidity of the drying air, rate of air flow, drying time and moisture content of the grain. The higher the initial moisture content of the grain and the longer the drying time, the greater is the danger of damage from a high drying temperature. At high moisture, high temperature may virtually cook the grain. So artificial drying requires a high degree of control to obtain the desired results. The problem of drying temperature is complicated because the farmer usually has only a general idea of the temperature of the air that passes through the grain and no estimate of the temperature of the grain itself. Braterskii (3) studied heat sensitivity of proteins of corn as a function of moisture, temperature and duration of heating. Below 40°C. no change occurred; complete denaturation occurred at 57.5°C. with 57.9% moisture within 30 minutes; with 26.6% moisture content at 60°C. and with 13.6% moisture at 70°C. the proteins were affected within 60 minutes.

The amount of loss of lysine during drying depends on

moisture content, temperature and rate of drying as was reported by Debczynska (4). Losses were larger at higher drying rates. In wheat containing 17 to 18% moisture content and dried to 13.5% at any temperature up to 80°C., or in wheat containing 23 to 24% moisture content and dried at 60°C., the losses in available lysine were small. A single-step lowering of moisture by 2 to 6% from about 23% reduced available lysine 3.5 to 7.5% at drying temperatures of 60 to 140°C. The available lysine was reduced by 28 to 42% if moisture was lowered by 4.9 to 9.4% in a single step. None of the drying procedures affected total lysine.

If corn is to be used for feeding, drying temperatures in the range of 95° to 135°F. are recommended (5). This is the grain temperature itself, not the air temperature. The air temperature may be higher than the grain temperature. As long as moisture is evaporating from the grain, the temperature of the grain will be lower than that of the drying air. Higher temperatures are said to decrease the feeding value of the protein (2). The damage observed after drying at temperatures below 95°F. was possibly due to molding during very slow drying. According to reports by the U.S.D.A. (5), temperatures below 95°F. decreased the lysine and tryptophan contents of corn as shown by studies with rats and swine.

The maximum air temperature for safe drying of seed corn is 110°F. (6). If the initial moisture content of the grain is below 25%, a temperature of 120°F. can be used (7).

For complete suitability, corn that is to be used for starch production by the wet-milling process should not be dried at temperatures above 140°F. (8). At higher drying temperatures the protein apparently is not only denatured but it is difficult for the miller to separate the starch from the protein. As a result, many complications arise during the milling process.

Watson and Sanders (9) studied the steeping characteristics of thin sections of the horny endosperm of corn kernels and found that sections of grain damaged by overheating during drying retained more starch. Cox et al. (10) showed that the SO₂ steep disperses the protein matrix in which the starch granules lie embedded in the endosperm cells. Overheating makes the protein harder to disperse, so the starch is not completely released from the matrix.

MacMasters et al. (11) reported corn dried in air at 200°F. gave starch in low yield, with high protein content. Corn dried at 180°F. gave low starch yield in most years and under most conditions. The 160°F. drying temperature had a damaging effect in a few cases. For safety it was recommended that grain not be allowed to reach a temperature above 140°F. Grain temperatures were not measured throughout the drying cycle in these experiments but the authors stated that the corn probably reached the temperature of the drying air. Kaufmann (12) suggested that no part of a lot of grain be heated above 140°F. or be dried below 13.5% moisture content.

No definite information is available as to what temperature is safe for drying corn that is to be used for dry milling. But the dry corn milling industry much prefers to use corn that has dried naturally rather than grain that has been artificially dried. The reason is that rapid drying causes strains and stresses that lead to cracking so that too much flour is obtained.

The safe temperature for drying corn for use in fermentation for alcohol production is much the same as that for drying seed corn. If the corn has been dried at too high a temperature, the alcohol yield is definitely decreased.

Adams et al. (1) reported that kiln drying of corn gave a 2 to 3% decrease in alcohol yield. When the drying temperature is above 200°F. the decrease in alcohol yield is 4 to 6%. The action of heat on the fermentable carbohydrate portion of the grain has not been definitely characterized but is probably a formation of a certain amount of unfermentable dextrins. Another possible factor is the inability of water and enzymes to penetrate through the protein matrix after it has been overheated.

The safe maximum temperature for drying wheat is probably the same as that recommended for drying corn. In Canada, the maximum air temperature allowable for drying wheat is 180°F., while in England it is recommended that the temperature be not over 150°F. (13, 14). A difference in the average relative humidity of the atmosphere in the two countries

probably accounts for the difference in allowed maximum temperature of drying air. Higher drying temperatures decrease the baking quality of the flour.

When the initial moisture content of the corn was less than or equal to 25%, no significant reduction in germination occurred at temperatures as high as 120°F. according to Wileman and Ullstrup (7). DeOng (15) obtained normal germination from corn exposed for 8 hours to a temperature of Little damage to germination of corn with 22.5% initial moisture content was found by Dimmock (16) when the grain was dried for a 12-hour period at temperatures up to 130°F. Dimmock said that some damage occurred at 130°F. when initial moisture was 26.0% and the duration of heating was 12 hours. When tested in the greenhouse the final germination was quite high but considerably delayed. McRostle (17) reported on corn dried on the stalk in the field to around 30% moisture. No appreciable damage was caused by the use of drving temperatures up to 130°F. when this corn was subsequently artificially dired on the ear. However, when the initial moisture content was over 50%, damage was evident at all drying temperatures over 105°F.

Effect of Overdrying

When corn has been dried too fast cracks and fissures form in the outer part of the kernel and on dry milling the endosperm yields small grits and considerable flour, both of

less value than large grits (8). Any treatment that causes death and breakdown of the germ renders corn difficult to degerminate, and also makes it hard to separate the oil from the germ. Oil from dead germ is of less value, because it contains a higher percentage of free fatty acids than is present in high-grade corn oil.

Statements have frequently been made that corn dried at high temperature is not suitable for use in starch production (8, 10, 11,18-21). Milling is incomplete and much endosperm, normally broken to free the starch, remains intact as corn grits that go to the feed house. Gelatinized starch when present in even small quantities in a wet milling plant can cause serious trouble because of its tendencies to plug screens or other small openings and to interfere with separation of normal starch. The dead germ is difficult to remove completely, and recovery of oil is also difficult. The oil is of poor grade with a high content of free fatty acids.

Reports on the decrease in nutritive value when corn is heated seem to differ among authors (Emerich et al., 22; Hathaway et al., 2; Jensen et at., 23; Liener 24). Many publications have shown drying with air at high temperatures to have little significant effect on the nutritive value of the corn, so it must be emphasized that grain temperature, not air temperature, is of influence. Workers who found a decrease in nutritive value based their conclusions on data

concerning actual grain temperature.

McGuire and Earle (18) reported the solubility of corn protein in water and in 0.01 N potassium hydroxide solution at 23.9°C. to decrease continuously with increasing drying temperatures of the grain from 120° to 200°F. (48.9° to 93.3°C.). With increasing drying temperatures denaturation or other changes in the physical state of the protein take place. But there was no report by these authors of any critical damage occurring at any particular temperature.

Many attempts have been made by various investigators to find a property of corn that is affected by drying and which could be adopted as the basis of a quick test for assaying damage done to corn by excessive heat. However, none of the work reviewed has indicated any property that appears to be adaptable to give an accurate and rapid method for practical use. Tests for glutamic acid decarboxylase activity (25), viability and germinability (26) have been used to measure the effects of artificial drying and storage. Heusdens and MacMasters (27) reported results of studies of methods for the determination of glutamic acid decarboxylase activity, viability, germination, mold count and fat acidity of dried and stored samples to determine changes in corn stored with little or no contamination with storage molds. Glutamic acid decarboxylase activity was the most satisfactory method to detect overheated corn, while mold count and fat acidity were good indices of changes in storage.

Extraction and Fractionation of Corn Proteins

Different proteins have been classified according to their solubility in various solvents. The albumins are soluble in water, globulins in saline solution, prolamines in alcohol and glutelin in alkali solution. The amount of each fraction obtained from a given source depends upon the method of extraction and the strength of reagent used.

Early work by Chittenden and Osborne(28) showed the corn kernel to contain several distinct proteins well characterized by their reactions and composition: three globulins (salt soluble), one or more albumins (water soluble) and an alcohol-soluble protein. Osborne and Mendel (29) found that 22% of the total corn protein was soluble in 10% potassium chloride, 41% in 90% alcohol, 31% in 0.2% potassium hydroxide solution, and 6% insoluble in all and therefore lost in the discarded residue. Of the total nitrogen of corn, 5.27% was reported by Spitzer (30) to be present as amide, 21.6% as globulin (soluble in 10% sodium chloride solution), 29.4% as zein (soluble in 90% alcohol), and 42.85% as glutelin.

Similar studies were made by Zeleny (31). He extracted the proteins from corn using water, molar sodium chloride solution, hot 80% ethyl alcohol, and 0.2% potassium hydroxide solution. Successive extractions yielded 6.28, 7.82, 41.97, and 16.92% of the protein, respectively, and 27.01% as nitrogen not peptized. Zeleny said part of the residue

consisted of seed coat proteins. Since these seed coat proteins are bound with insoluble carbohydrate they are very difficult to separate.

By using various modifications of the classical Osborne method (29), Nagy et al. (32) and Jimenez (33) studied extraction of corn proteins. These workers used dilute salt solution to remove albumins and globulins, aqueous ethyl alcohol solution to extract zein and dilute alkali solution to separate glutelins. The recovery of total soluble protein was not more than 80 to 90%.

An alternative approach to fractionation of the protein is that developed by Mertz and co-workers (34, 35). By using an alkaline medium containing sodium, copper, sulfate and sulfite ions, Mertz and Bressani (34) obtained 95.3% extraction of the endosperm nitrogen of corn and 97.9% of the germ nitrogen. For comparison, the same corn endosperm samples were also extracted by a modified Osborne method (32) and gave a yield of 83.1% of the total nitrogen, 12.4% extracted by the use of 5% potassium chloride, 33.9% extracted by 71% ethanol and 36.8% dissolved in 0.2% sodium hydroxide.

Later studies were made by Mertz et al. (35). These workers further fractionated the alkaline copper extracts of the endosperm from a U. S. and a Guatemalan sample of corn into three fractions: an acid-soluble fraction, an alkaliand alcohol soluble fraction (zein) and an alkali-soluble, alcohol-insoluble fraction (glutelin). The three fractions

accounted respectively for 23.4, 46.9 and 24.3% of the endosperm nitrogen in the U.S. sample, and 24.3, 44.7, and 21.0% of the endosperm nitrogen in the Guatemalan sample.

The research reported (28-35) indicated considerable disagreement as to the distribution of the various proteins in corn. Nagy et al. (32) studied the factors affecting the solubility of corn proteins and pointed out that protein fractionation of corn by using different extraction solvents has proved to be inaccurate as a quantitative procedure, since large variations in the percentage of the different fractions extracted are caused by slight alterations in the procedures such as fineness of grinding, length and temperature of extraction, presence of salts, etc. Nevertheless these authors considered the alcohol-soluble fraction to be the most well defined of all and the one least affected by slight variations in experimental techniques and reported that addition of 0.5% sodium acetate in the 85% ethanol as a buffer improved extraction of zein and gave more consistent results, presumably by counteracting adsorption.

As reported by Nagel et al. (36) the ratio of water to meal, temperature and the time of extraction were not critical for the extraction of soybean protein. But the size of meal particles was found to be a very important factor. Meal ground to pass through a 100-mesh screen was reported to be satisfactory for the purpose of extracting nitrogenous compounds.

Some studies have shown that the proportions of the salt-, alcohol-, and alkali-soluble proteins present in corn vary with the type or strain of corn and the age of the kernel (30, 31, 37, 38).

Showalter and Carr (37) reported that in corn of low protein content both amides and albumins seem to form a considerably greater proportion of the total protein than in corn of high protein content. The proportional content of zein to total protein is greater as the total protein content of corn increases. This increase in the proportion of the zein seems to be accompanied by a corresponding decrease in the proportion of the glutelin. Total nitrogen content apparently is correlated with the proportions of various proteins. These same authors reported that, when 90% alcohol was used as the extracting agent, 8.1% zein was found in corn containing 15.7% protein and 2.2% zein in corn with 8.0% protein.

The relative proportion of the various proteins of the corn kernel at different stages in its development was studied by Zeleny (31). Zeleny concluded the globulin and glutelin are synthesized at a relatively uniform rate throughout the growth period. Zein content is low in the very immature corn kernel but as the corn approaches maturity zein is synthesized at a very rapid rate. The rapid increase of zein as percentage of total nitrogen is almost exactly paralleled by the decrease in water-soluble non-protein nitrogen. Hansen et al. (39) reported the zein in 18 samples was linearly related to

total protein content ranging from 6.3 to 19.7%.

In the study of protein and amino acid content of different corn varieties, Bressani and Mertz (40) extracted the proteins of the germ and endosperm of U. S. and Guatemalan selections with an alkaline copper reagent and separated each of the extracts into three fractions. The results showed that alcohol-soluble nitrogen of endosperm increased with increasing crude protein content of the whole grain.

In the endosperm samples, the acid-soluble proteins comprised from 17 to 26%, the alkali- and alcohol-soluble proteins, zein, from 41 to 60%, and the alkali-soluble, alcohol-insoluble proteins, glutelin, from 17 to 31% of the total nitrogen. Corn germs similarly fractionated yielded 30 to 40% acid-soluble, 5 to 10% alkali- and alcohol-soluble, and 49 to 54% alkali-soluble, alcohol-insoluble proteins as percentage of the total nitrogen.

The nutritional quality of corn germ protein has been recognized as approximating that of animal proteins, both from feeding experiments and comparative amino acid analysis by Block and Bolling (41) and Mitchell and Beadles (42). The low nutritional value of zein has been studied by numerous workers including Willcock and Hopkins (43) and Osborne and Mendel (29). These workers demonstrated that on a diet in which the only source of protein was the alcohol soluble protein, i.e., zein, the animals lost weight. Thus zein was demonstrated to be nutritionally incomplete. But by the addition of lysine and tryptophan to the diet normal growth

was restored. Zein was reported to be absent from corn germ although it constitutes the major protein of the endosperm. The rest of the endosperm protein, largely glutelin, was shown to contain the amino acids that zein lacks. Mitchell et al. (44) said the proportion of tryptophan and lysine in the total protein of corn decreased with increasing protein content.

As compared to ordinary dent corn, corn high in lysine is low in alcohol-soluble protein (33, 45-47). Nelson (47) found that zein of ordinary dent corn comprises 54.2% of the total soluble protein of the endosperm. Zein amounts to only 25.4% of the total soluble protein in the endosperm of Opaque-2 corn, and for Floury-2 corn the comparable value is 28.7%.

The state of aggregation of proteins is changed by heat treatment and this increases the resistance to any peptizing action (48). The more severe the heat treatment, the greater the increase in resistance, giving a distinctly decreased solubility.

McGuire and Earle (18) studied changes in the solubility of corn protein resulting from the artificial drying of high-moisture corn. The initial moisture contents were 20 and 30%, and the grain was dried to 12% moisture content at several temperatures between 120° and 200°F. Then successive extractions were made with water, 5% sodium chloride, 60% ethyl alcohol at 175°F, and 0.2% potassium hydroxide from both a

sample dried at 200°F. and an air-dried control. The results were: albumin 12.2% for the sample dried at 200°F. and 15.8% for the air-dried sample, globulin 3.7% and 7.2%, prolamine 40.7% and 38.3%, glutelin 17.2% and 19.2%, respectively. These data indicate the greatest differences were found in the water-soluble and 5% sodium chloride-soluble fractions.

As the time and temperature increased in the autoclaving of corn flour, greater losses of albumin, globulin, prolamine, and glutelin (49) occurred. Nonpeptized and nonprotein nitrogen contents increased under similar treatment.

In a study of gluten, Sharp and Gortner (50) found that the glutenin fraction is more sensitive to heat than the gliadin fraction. The same conclusion was reported by Cook (51) from viscosity and solubility measurements on gluten dispersions.

Mori (52) reported that during storage of corn, the protein soluble in water and neutral salts decreased but that which was soluble in dilute alkalies increased, or glutelins increased at the expense of globulin. During the change a part of the globulin disintegrated, increasing nonprotein nitrogen content, and simultaneously acidic decomposition products were formed so that pH of the extract was lowered from 6.5 to 4.

MATERIALS AND METHODS

Sample Preparation

In the fall of 1968 yellow dent hybrid corn variety 60SC was harvested after drying in the field at Ashland Bottoms test farm plots, Manhattan, Kansas. After shelling, the corn was cleaned with an aspirator. The grain was divided into three sets of samples which were dried at air temperatures of 120°F., 140°F., and 180°F., respectively, in an air oven from an initial moisture content of 15.1% to final moisture contents of 12.4%, 10.2% and 10.2%, respectively (Table I).

After being scoured, the corn was tempered for two hours to a moisture content of 16%, and then milled on a Ross mill to yield two flours, one that passed through a 62-mesh sieve, and the other remaining on the sieve. The milling process included first break, second break, sizing, germ, grader, and four reduction rolls. The fine meal, "thru 62 mesh", was primarily floury endosperm. The coarse meal, "over 62 mesh", was primarily horny endosperm. The flow sheet of the Ross mill is shown in Fig. 1.

Whole corn meal was produced by grinding the grain in a Mikro-Sample Mill to pass through a screen size of 0.027 in. Each uniformly mixed sample was stored in a cold room (40°F.) in an air-tight container.

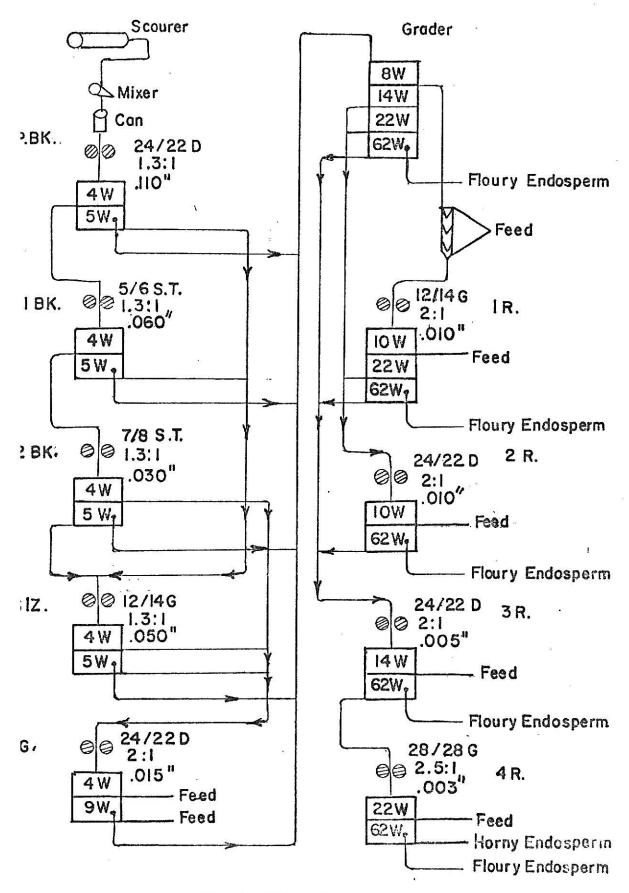


Fig.I. The Ross Mill flow sheet.

TABLE I. DRYING CONDITIONS

Air Temperature °F.	Initial Moisture %	Final Moisture %	Drying Time
120	15.1	12.4	4 hr.
140	15.1	10.2	3 hr. 30 min.
180	15.1	10.2	1 hr. 10 min.

Proximate Analysis

Using A.O.A.C. methods (53) the proximate composition of each of the ground grain samples was determined. Aliquots of the respective protein fractions were analyzed for nitrogen by the micro-Kjeldahl procedure (53). The nitrogen content of the residue was determined by the conventional Kjeldahl method (53). Percentage of nitrogen was multiplied by the factor 6.25 to obtain the percentage of crude protein.

Protein Fractionation Procedures

The procedures of Osborne and Mendel (29, 54) as modified by Nagy et al. (32), with further minor modification by Skoch (55) was used for fractionation and extraction. Samples of whole corn meal, horny endosperm and floury endosperm from corn dried at different temperatures were used as raw materials from which to extract the protein by successive use of each of the following solvents: distilled water to

extract the albumin, 5% sodium chloride solution for globulin, 80% ethanol plus 0.2% sodium acetate solution for prolamines, and 0.2% sodium hydroxide solution for glutelins. The residual proteins (scleroproteins) were retained for analysis.

Water Extraction. To extract water-soluble proteins (albumin), a 3-g. sample was transferred into a 250-ml centrifuge tube and 100 ml. of distilled water was added. The stoppered tube was rotated longitudinally for 24 hr. at room temperature. Suspensions were then centrifuged at 2000 r.p.m. in an International No. 2 centrifuge for 20 min. and the supernatant decanted. Twenty-five ml. of distilled water was added to the material in the centrifuge tube and the mixture was stirred with a glass rod to resuspend the precipitate and then centrifuged. The precipitate was washed twice by this procedure. The supernatant liquid was frozen at 4°C. until it was analyzed for protein content.

Salt Extraction. The precipitate from the water extraction was combined with a 5% (w/v) solution of sodium chloride to extract salt-soluble protein (globulin) by the same procedures as used to extract albumin. Prior to analysis for protein content of this fraction, the supernatant was dialyzed continuously for 72 hr. against four changes of distilled water at 40°F. then tested with silver nitrate to verify complete removal of salt.

Alcohol Extraction. A mixture of 18.8% water, 80% absolute ethanol, and 0.2% sodium acetate (v/v/w) was used in extracting the insoluble residue with the same extraction

procedures used for the prior two solvents. Aliquots of the sample were used for estimating crude protein content.

Alkali Extraction. Following successive extraction with distilled water, sodium chloride solution and ethanol-acetate solution, the insoluble residue was similarly extracted with 0.2% (w/v) sodium hydroxide solution. Aliquots of the supernatant liquid from the alkali extraction were used for analysis.

Insoluble Residue. The wet residue after extraction with the four different solvents mentioned above was dried in an air oven at 50°C. for 24 hr. The dried residue was quantitatively recovered, weighed and analyzed for crude protein by the macro-Kjeldahl method (53).

Amino Acid Analysis of the Total Soluble Protein

Amino acid analysis of the acid hydrolyzed sample was made following the procedure of Spackman et al. (56) supplemented by that of Moore et al. (57) for cystine. These analyses were made by means of a Beckman 120 automatic amino acid analyzer.

Aliquots of each of the four fractions of the whole corn meal from corn dried at 120°F. were combined in the ratios in which they were obtained and evaporated to reduce the volume. The same was done with aliquots from corn dried at 180°F. Five ml. of condensed solution were placed in a 15 x 150-mm. narrowed test tube. Five ml. of concentrated

hydrochloric acid were added. The tube was placed in dry ice-alcohol bath, frozen, and attached to an aspirator. The test tube was sealed in vacuum of $27\frac{1}{2}$ in. mercury. Then the sealed tube was put in an oven at 110° C. for 22 hours. Hydrolyzed samples prepared for analysis were stored at -20° C, until used. Humin was removed by filtering each hydrolyzed sample through a fritted disc funnel. The filtrate was evaporated to dryness three times under reduced pressure, redissolved in distilled water after each of the first two evaporations and finally made up to a known volume with 0.2 N sodium citrate buffer, pH 2.2.

RESULTS AND DISCUSSION

Yield of Milled Corn Products and Their Chemical Composition

The drying conditions for the corn samples are given in Table I. The yields of milled corn products obtained by milling the dried grain on the Ross Mill are shown in Table II. Portions of the same corn sample dried at different temperatures at the same time were ground at two different times as represented by A and B. Variation in yield of any one fraction from the samples dried at the three different temperatures was not great. For the horny endosperm (over 62 mesh) the percentage yield averaged 27.8% for A, and 37.3% for B at all three different temperatures. For floury endosperm (through 62 mesh) the average percentage yield was 48.4% for A and 33.5% for B. The average yield of "overs" and "thrus", 62 mesh, was 76.2% for A and 70.9% for B. For comparison it may be noted that the percentage extraction used in dry corn milling is usually about 70%.

TABLE II. YIELD OF MILLED CORN PRODUCTS

Air		Endosperm	Floury	Endosperm	Total	Yield
Temperature °F.	(over	62 mesh) %	(thru	62 mesh) %	%	
	A	В	A	В	A	В
120 140 180	25.8 29.5 28.0	37.0 37.9 37.1	49.2 48.6 47.4	32.5 34.8 33.3	75.0 78.1 75.5	69.6 72.8 70.4
Average	27.8	37.3	48.4	33.5	76.2	70.9

Little important difference in the chemical composition of the corn grain and corn flour resulted from drying at any of the various temperatures as determined by the use of AOAC methods No. 2.044, 13.003, 13.006, 22.032, 22.033, 22.034, 22.042 (53) (Table III).

Only the protein content of the meal from whole corn which was dried at different temperatures showed a significant difference. When corn was dried at 120°F. as control the meal had a protein content of 10.5%. Meal from corn dried at 140°F. had a protein content of 9.8% and that of meal from corn dried at 180°F. was 9.4%. There was a tendency for the protein content to decrease as the drying temperature increased from 120°F. to 180°F. This was unexpected and in disagreement with previous data of other workers.

For the macro-Kjeldhl method (53) the digestion time was increased to three different time intervals; one hour, two hours and four hours. The results are shown in Table IV. The results showed the difference in protein contents to result from differences in the samples dried at different temperature not from the digestion time used. For the A.O.A.C. method (53), one hour digestion time will detect all the nitrogen in the sample except that in nitrate and nitrite form.

To determine whether the differences in protein content actually resulted from differences in drying or were the result of errors in sampling, a field-dried, unheated corn sample was used to repeat the experiment.

TABLE III. CHEMICAL COMPOSITION OF CORN AND MILLED CORN PRODUCTSa

Sample	Moisture %	Crude Fat %	Crude Fiber %	Ash %	Protein %
Whole corn meal					
120°F. 140°F. 180°F.	12.1 10.3 9.9	5.2 5.0 5.3	2.8 2.4 2.3	1.5 1.6 1.5	10.5 9.8 9.4
Horny endosperm	a e				
120°F. 140°F. 180°F.	14.0 14.2 14.5	3.3 2.5 2.6	1.5 1.3 1.3	1.1 1.0 0.8	11.8 10.5 10.4
Floury endosperm	1	\$X			
120°F. 140°F. 180°F.	14.7 14.8 14.4	1.9 2.0 1.7	0.9 0.8 0.7	0.6 0.5 0.4	8.0 7.2 6.9

^aAll data reported on dry basis.

TABLE IV.	PROTEIN CONTENT USING TH	REE DIFFERENT DIGESTION
	TIMES IN THE A.O.A.C. ME	THOD (2-044)

Sample	Digestion Time hr.	Protein Content % d.b.
1	1 2 4	10.2 10.1 10.1
2	1 2 4	9.1 9.0 8.9
3	1 2 4	9.3 9.1 9.3

The corn sample was tempered with sufficient water to raise the moisture from 11.8% to 15.3% in a can kept in the refrigerator for 48 hours. Then this sample was divided into two parts. One part was dried at 120°F. in the air oven for 3 hours to a final moisture of 11.8%. The other part was dried at 180°F. for 0.75 hours to a final moisture content of 11.1%.

By using a Mikro-Sample Mill these two samples were ground to pass through a screen size of 0.027-inch. The protein contents were determined by the macro Kjeldahl (53) method. For the corn samples dried at 120°F. and 180°F. the protein contents were 8.2% and 8.0% (on dry basis), respectively.

Protein fractionation of these two corn samples was carried out and the results are shown in Table V.

Compared with the fractionation of protein from whole

TABLE V. RESULTS OF THE FRACTIONATION BY THE MODIFIED OSBORNE-MENDEL METHOD OF PROTEIN FROM WHOLE CORN MEAL OF CORN DRIED AT 120°F. and 180°F.

Fraction ^a	120°F.	180°F.
Albumin	6.6	7.9
Globulin	9.5	8.8
Prolamine	18.6	16.0
Glutelin	42.7	43.8
Total soluble protein	77.4	76.5
Insoluble residue	20.5	16.7
Total protein recovered	97.9	93.2
Initial protein of grain, (%)	7.2	7.1

aData reported as percentage of total protein.

bEach value is the average of results of two protein fractionations.

corn meal of corn dried at 120°F., 140°F. and 180°F. in Table X, the distribution of each fraction within one corn sample or the tendency toward increase or decrease of the protein percentage of each fraction due to heat treatment were the same as in the earlier work. The earlier differences in protein content therefore were not the result of differences in drying, but of sampling errors.

In fact, since the results of protein fractionation were reported as percentage of total protein in the original sample, the differences in protein content of each sample were of no significance.

Results of Protein Fractionation

In studying differences in the solubility of protein of artificially dried corn samples of 120°F., 140°F. and 180°F., preliminary comparisons were made on the effect of particle size on the protein extraction.

The floury endosperm from corn dried at 120°F. was used in this study. Fifty g. of floury endosperm was sieved in a Tyler sieve; 28.8 g. of the sample passed through the 100-mesh sieve and was removed. The over-100-mesh sample was divided into two parts; A and B. The particle size of A was classified again as over 60-, 70-, 80- and 100-mesh (Tyler sieve size). The results were recorded and the fractions remixed carefully. Sample B was reground finely enough to pass through 100 mesh. Protein fractionation was carried out separately on A and B. Both the particle size classification and protein fractionation

results are shown in Table VI. The data showed slightly larger amounts of soluble protein could be extracted from the flour passing through a 100-mesh sieve. Reduction in particle size increased the protein extractability. This observation was also made by Nagel et al. (36) while extracting protein from soybean meal and by Smith (58) and Nagy et al. (32) in working with corn. But, since this difference is not significant for the extraction method used, the effect of particle size on protein extraction was neglected.

The results of protein fractionation from the whole corn meal, horny endosperm and floury endosperm extracted with each of the solvents and reported as percentage of total protein are given in Tables VII, VIII, and IX. The tables also include the protein content of the original meal and the percentage of insoluble protein remaining after the extraction process. The percentage of protein remaining in the insoluble residue was added to the total soluble protein to give the total protein recovered as a check on the method. The total protein recovered was within normal limits of experimental error. Values in each case were averaged and the following discussion of results is based upon the average values.

Results of successive extractions made with distilled water, 5% sodium chloride solution, 80% ethanol plus 0.2% sodium acetate solution and 0.2% sodium hydroxide solution on samples of whole corn dried at 120°F., 140°F. and 180°F.

TABLE VI. EFFECT OF PARTICLE SIZE DISTRIBUTION ON PROTEIN FRACTIONATION

1. PARTICLE SIZE DISTRIBUTION OF SAMPLES Aa and Bb

Particle Size	Samples (g.) A B	
Over 60-mesh Over 70-mesh Over 80-mesh Over 100-mesh Thru 100-mesh	0 0 1.90 0 4.75 0.08 3.53 0.67 0 8.17	

2. RESULTS OF THE PROTEIN FRACTIONATION FROM CORN SAMPLES A AND B BY THE MODIFIED OSBORNE-MENDEL METHOD

Fraction ^c	Sample A	s B
Albumin Globulin Prolamine Glutelin	2.2 5.0 25.7 45.2	1.7 3.1 27.5 49.6
Total soluble protein	78.1	81.9
Insoluble residue	10.6	11.7
Total protein recovered	88.7	93.6
Initial protein of grain (%)	8.4	8.6

 $^{^{}a}A$ = the sample over 100-mesh of whole corn meal from corn dried at 120°F. ^{b}B = the sample over 100-mesh of whole corn meal from

dried at 120°F. and reground to pass through 100-mesh.

^cData reported as percentage of total protein.

TABLE VII. COMPLETE RESULTS OF THE FRACTIONATION BY THE MODIFIED OSBORNE-MENDEL METHOD OF PROTEIN FROM WHOLE CORN MEAL OF CORN DRIED AT 120°F., 140°F. and 180°F.

Fraction ^a	120	°F.	140°	F.	180°F	•
	Ap	Вр	A	В	A	В
Albumin	5.7	5.9	6.4	6.7	6.6	6.8
Globulin	8.3	8.9	6.8	7.8	7.4	7.2
Prolamine	24.0	30.4	19.0	20.0	19.2	21.2
Glutelin	52.6	48.2	52.5	46.7	50.5	47.2
Total soluble protein	90.6	93.4	84.7	81.2	83.7	82.4
Insoluble residue	13.2	9.6	14.2	11.3	14.8	10.8
Total protein recovered	103.8	103.0	98.9	92.5	98.5	93.2
Initial protein of grain (%)	9.3	9.0	8.6	8.0	8.3	8.2

^aData reported as percentage of total protein

^bA and B represent corn ground at different times but harvested and dried at the same time.

COMPLETE RESULTS OF THE FRACTIONATION BY THE MODIFIED OSBORNE-MENDEL METHOD OF PROTEIN FROM HORNY ENDOSPERM OF CORN DRIED AT 120°F., 140°F. and 180°F. TABLE VIII.

Fractiona		120°F.	°F.			140°F.	°F.			180°F.	°F.	
	АЪ		qB		A			В	A		В	onusta
Albumin	3.5	3.0	2.3	3.9	2.3	3.0	1.7	6.0	2.0	2.2	1.4	0
Globulin	6.4	6.3	8.5	7.7	7.3	5.6	5.5	7.0	5.8	4.8	5.0	5.0 3.6
Prolamine	36.9	35.5	38.9	36.5	29.8	28.9	33.0	32.2	32.1	29.6	33.6 33.6	33.6
Glutelin	5.44	44.0	4.5.4	7.97	48.9	6.64	45.7	4.7.4	6.44	47.8	51.4 50.5	50.5
Total soluble protein	9.46	88.6	95.1	94.3	88.3		86.8 85.6	87.5		84.8 84.4 91.4 87.7	91.4	87.7
Insoluble residue	10.2	12.0	15.8	15.8 10.8	12.6	14.1	12.6 14.1 11.3 10.9	10.9		12.0 12.6 12.3 11.8	12.3	11.8
Total protein recovered	104.8	104.8 100.6	110.9	10.9 105.1	100.9	100.9 100.9	6.96	7.86	96.8	97.0	97.0 103.7 99.5	99.5
Initial protein of grain (%)	10.1		ъ. Ф	40	0.6	0	2	7.6	₩	8.9	7.2	ત્ય

 $^{\mathrm{a}}\mathrm{D}$ ata reported as percentage of total protein.

 $^{\text{b}}\text{A}$ and B represent corn ground at different times but harvested and dried at the same time.

COMPLETE RESULTS OF THE FRACTIONATION BY THE MODIFIED OSBORNE-MENDEL METHOD OF PROTEIN FROM FLOURY ENDOSPERM OF CORN DRIED AT 120°F., 140°F. and 180°F. TABLE IX.

	30 30	33 35 35										
Fraction ^a		120°F.	·F.			140°F.	°F.			180°F.	°F.	
	Аb	_	дE		A			В	A		В	
Albumin	2.6	2.0	3.5	1.6	1.3	1.1	1.3	1.3	1.0	1.2	0	1.3
Globulin	8.4	5.5	9.0	3.7	5.8	5.0	5.0	3.8	4.1	4.1	0.4	2.4 0.4
Prolamine	27.0	27.5	26.3	30.0	26.7	22.2	21.3	23.1	23.7	24.1	23.3	25.3
Glutelin	57.4	56.5	53.2	56.8	59.9	58.3	7.65	7.65	62.7	0.09	60.0 62.7	62.7
Total soluble protein	4.56	91.5	91.7	92.1	95.7	96.6	87.0	87.6	91.5	4.68	87.3 94.0	0.46
Insoluble residue	12.0	12.0 10.0	10.5	10.0	13.3	12.8	7.6	10.0	10.0 14.7 13.5	13.5	9.3 9.3	9.3
Total protein recovered	107.4	107.4 101.5	102.2 102.1	102.1	109.0	7.66	7.96	9.76	97.6 106.2 102.9	102.9	96.6 103.3	103.3
Initial protein of grain	٠ <u>,</u>	t 0	7.9	-4	6.1	д	32	5.3	5.8	1 00	5.0	. 0

 $^{
m a}{
m Data}$ reported as percentage of total protein.

 $^{\mathrm{b}_{\mathrm{A}}}$ and B represent corn ground at different times but harvest and dried at the same time.

and on meal, horny endosperm and floury endosperm from the corn samples are shown in Tables X, XI, and XII. The relative proportions of fractions are reported as percentages of total protein.

The results of protein fractionation of the whole corn meal are shown in Table X. Each value is the average of results of two protein fractionations of two samples of corn dried at the same time but ground at different times.

The data were subjected to a one way analysis and also to the LSD test (59). For the albumin fraction the percentage of protein extracted increased as the temperature of drying increased. The amount of protein extracted increased from 5.8% for the control dried at 120°F. to 6.6% for the corn dried at 140°F. and 6.7% for the corn dried at 180°F. These data gave a significant difference for heat treatment. The LSD test showed the temperature of 120°F. had a significant difference from temperatures of 140°F. and 180°F. but there was no significant difference between 140°F. and 180°F.

The heat-damaged corn (dried at 180°F.) showed an increase of 16% in the albumin fraction when compared with the corn dried at 120°F. The globulin, prolamine and glutelin fractions showed no significant difference but there was a tendency for the protein in these fractions to decrease as the temperatures increased. The globulin fraction decreased from 8.6% for 120°F. to 7.3% for 180°F. and thus indicated a 15% protein loss when the corn was dried at 180°F.

TABLE X. RESULTS OF THE FRACTIONATION OF PROTEIN^a, ONE WAY ANALYSIS AND THE LSD TEST^b ON WHOLE CORN MEAL FROM CORN DRIED AT 120°F., 140°F. and 180°F.

Fraction	120°F.	140°F.	180°F.	Loss% ^c	
Albumin	5.8	6.6	6.7	-16	s
Globulin	8.6	7.3	7.3	15	n.s.
Prolamine	27.2	19.5	20.2	26	n. s.
Glutelin	50.4	49.6	48.9	3	n. s.
Total soluble protein	92.0	83.0	83.1	10	S

^aData reported as percentage of total protein.

^bValues lying above the same horizontal line are not significantly different in the LSD test; those over different lines are with $\alpha = 0.05$.

CLoss, % = (percentage extracted from corn dried at 120°F. - percentage extracted from corn dried at 180°F.) / percentage extracted from corn dired at 120°F. multiplied by 100.

TABLE XI. RESULTS OF THE FRACTIONATION OF PROTEIN^a, ONE WAY ANALYSIS AND THE LSD TEST^b ON HORNY ENDOSPERM FROM CORN DRIED AT 120°F., 140°F. and 180°F.

Fraction	120°F.	140°F.	180°F.	Loss %	c
Albumin	3.2	2.0	1.4	56	s
Globulin	8.1	6.3	4.8	41	s
Prolamine	36.9	31.0	32.2	13	s
Glutelin	45.0	47.8	48.7	-8	n. s.
Total soluble protein	93.2	87.1	87.1	7	s

^aData reported as percentage of total protein.

bValues lying above the same horizontal line are not significantly different in the LSD test; those over different lines are with $\alpha = 0.05$.

TABLE XII. RESULTS OF THE FRACTIONATION OF PROTEIN^a, ONE WAY ANALYSIS AND THE LSD TEST^b ON FLOURY ENDOSPERM FROM CORN DRIED AT 120°F., 140°F. and 180°F.

				9 35 500 STOR	
Fraction	120°F.	140°F.	180°F.	Los	s % ^c
Albumin	2.4	1.3	0.9	63	S
Globulin	6.7	4.9	4.2	37	n. s.
Prolamine	27.7	23.3	24.1	13	S
Glutelin	56.0	59.3	61.4	-10	s
Total soluble protein	92.7	89.2	90.6	2	n. s.

aData reported as percentage of total protein.

bValues lying above the same horizontal line are not significantly different in the LSD test; those over different lines are with $\alpha = 0.05$.

CLoss, % = (percentage extracted from corn dried at 120°F. - percentage extracted from corn dried at 180°F) / percentage extracted from corn dried at 120°F., multiplied by 100.

The prolamine fraction decreased from 27.2% to 20.2%. a total of 26% decrease. There was a decrease from 50.4% to 48.9% or a loss of 3% for the glutelin fraction of whole corn meal. Finally, the percentages of albumin, globulin, prolamine and glutelin extracted from the whole corn meal were added to give the percentage of total soluble protein. The total soluble protein was 92.0% for the control dried at 120°F., 83.0% for the corn dried at 140°F. and 83.1% for corn dried at 180°F. There was therefore a 10% loss when corn was dried at 180°F. compared with the control. The one way analysis showed a significant difference for the heat treat-As the temperatures increased, the protein solubility decreased and the total protein from corn dried at 120°F. was significantly different from that of corn dried at 140°F. and that of corn dried at 180°F. But between the corn dried at 140°F. and that dried at 180°F. there was no significant difference. This means as the drying temperature was increased above 140°F. there were some physical and chemical changes occurring inside the corn kernel, but the changes were slight and uniformly affected the four protein fractions. The changes were so small as not to be statistically significant but as they were added the effect of heat damage became apparent.

The results of protein fractionation of the samples of horny endosperm are shown in Table XI. Each datum was from the average of four different protein fractionations.

The data indicated that all samples had a larger percentage of the glutelin fraction than of the three other soluble fractions; the percentage of glutelin varied from 45.0% to 48.7%. As the temperatures increased, the glutelin solubility increased and when the corn had been dried at 180°F. the gain was 8% as compared with the control, dried at 120°F. But the difference was not statistically significant when subjected to one way analysis.

The albumin fraction was the smallest among the four fractions extracted and changed from 3.2% for the control dried at 120°F. to 2% for the corn dried at 140°F. and to 1.4% for the heat damaged corn dried at 180°F. Thus heat damage resulted in a loss of 56% solubility of this fraction. The LSD test showed the protein solubility was significantly different for the corn dried at 120°F. and 180°F. Between samples dried at 120°F. and 140°F. or 140°F. and 180°F. there was no significant difference.

The solubility of globulin decreased as the temperature of drying increased; the loss was 41%. There was 8.1% protein extracted from corn dried at 120°F. 6.3% extracted from corn dried at 140°F. and 4.8% for the heat damaged corn. These data gave a significant difference on one way analysis. The LSD test showed the drying temperature of 120°F. had a significant difference from 180°F. Between 120°F. and 140°F. or 140°F. and 180°F. there was no significant difference for heat treatment.

The prolamine fraction was the second largest fraction extracted from the corn samples. It changed from 36.9% for the control dried at 120°F. to 31.0% for the corn dried at 140°F. and to 32.2% for the corn dried at 180°F. As the drying temperature increased the protein solubility decreased; the loss was 13%. The data gave a significant difference for heat treatment. The LSD test showed the temperature of 120°F. had a significant difference from temperature of 140°F. and 180°F., but there was no significant difference between 140°F. and 180°F. Glutelin and prolamine are known to be major protein fractions of corn endosperm (33, 40).

The total soluble protein also showed a significant difference depending on the heat treatment. As the temperature was raised the protein solubility of the horny endosperm decreased. For the control dried at 120°F. the total protein solubility was 93.2%; it decreased to 87.1% for the corn dried at 140°F. or 180°F. The decrease amounted to 7%.

The results of protein fractionation from the floury endosperm are shown in Table XII. Each value is the average of four different protein fractionation results.

These results were compared with the data obtained from protein fractionation of horny endosperm. There were less albumin, globulin and prolamine extracted than from horny endosperm, but the glutelin fraction in the floury endosperm was greater than that in the horny endosperm.

The amount of albumin fraction decreased as the temperatures increased. It changed from 2.4% at 120°F. to 1.3% at

140°F. and to 0.9% for heat damaged corn dried at 180°F. The total decrease amounted to 63% which was the highest change noted in this study. The reason is, the albumin was heated and so coagulated and became insoluble in water.

The globulin fraction showed no significant differences resulting from heat treatment when the data were subjected to one way analysis. There was a tendency toward a decrease as the temperatures increased from 120°F. to 180°F. There was 6.7% of globulin extracted from corn dried at 120°F., and 4.9% and 4.2% for corn dried at 140°F. and 180°F., respectively; the total decrease was 37%.

The prolamine fraction changed from 27.7% for corn dried at 120°F. to 23.3% and 24.1% for the samples dried at 140°F. and 180°F., respectively. One way analysis showed a significant difference between the heat damaged and the other samples; the total loss was 13%. The LSD test showed no significant difference between 140°F. and 180°F., but there was a significant difference between these and 120°F. as drying temperatures for the corn from which the floury endosperm was separated.

As the temperature at which the corn had been dried increased the solubility of the glutelin fraction increased. The total increase was 10%. When the corn had been dried at 120°F., the glutelin fraction was 56.0% of the total protein. For corn dried at 140°F. and 180°F. the glutelin solubility was increased to 59.3% and 61.4% respectively. The LSD test

also showed that glutelin separated from floury endosperm of corn dried at 120°F. was significantly different in amount from that of the fraction from corn dried at 140°F. and 180°F. There was no significant difference between the values for the fractions from corn dried at 140°F. and 180°F.

The total soluble protein of these floury endosperm samples showed no significant difference. There was a tendency, as the temperatures increased, for the protein solubility to decrease. The extraction was 92.7% for the corn dried at 120°F., 89.2% for corn dried at 140°F., and 90.6% for the corn dried at 180°F. The total decrease was 2%.

This study showed relatively small changes resulting from differences in drying temperature. If the corn had been dried from higher moisture content much larger differences might have been expected. Some workers have reported a reduction in protein solubility, feeding value and damage for wet and dry milling when corn was dried at high temperature (2, 8, 11, 18). These workers dried their corn samples from a moisture content of 20 to 30% to 10 to 12% moisture content, but in the present research the corn was dried from 15.1% moisture content to either 12.4 or 10.2% moisture content. Only 3% to 5% moisture was driven out by the heat treatment and therefore the corn was probably not damaged as much as when more drastically dried from 25% to 10-12% moisture content.

For horny endosperm, the total soluble protein showed a significant decrease resulting from heat damage. This seems

reasonable when one considers the structure of the corn kernel. The horny endosperm is mostly near the outer part and floury endosperm inside. Surface water is lost very quickly, but thereafter the drying rate is dependent on the relatively slow speed with which water will travel from the inside to the outside of the grain. Therefore the interior of the grain may be both hot and damp while the outside is hot but dry.

Although the total soluble protein of floury endosperm showed no significant change due to heat damage, there was a significant change in the albumin, prolamine and glutelin fractions. The albumin fraction decreased 1.5% from corn dried at 120°F. to corn dried at 180°F.; the prolamine decreased 3.6% from corn dried at 120°F. to corn dried at 180°F.; and the glutelin increased 5.4%. This may indicate that the protein was denatured and shifted from one fraction (albumin and prolamine) to the other (glutelin).

These results are consistent with results reported by other workers who have advised keeping corn temperature below 140°F. during drying.

Amino Acid Composition of the Total Soluble Protein

The amino acid analysis of the total soluble protein from the whole corn meal (Table XIII) showed no significant difference between the control (corn dried at 120°F.) and the most severely heat-treated corn sample (dried at 180°F.)

TABLE XIII. AMINO ACID COMPOSITION OF THE TOTAL SOLUBLE PROTEIN (EXPRESSED AS GRAMS AMINO ACID PER 100 GRAM PROTEIN)

Amino Acid	120°F.	180°F.
Lysine	1.91	2.02
Histidine	2.64	2.64
Arginine	0.72	1.89
Aspartic acid	6.39	6.55
Threonine	3.50	3.52
Serine	4.96	4.76
Glutamic acid	22.43	21.09
Proline	10.36	9.09
Glycine	4.00	4.00
Alanine	8.25.	8.36
Half cystine	0.00	0.00
Valine	4.88	5.02
Methionine	2.21	1.16
Isoleucine	3.63	3.71
Leucine	13.82	14.28
Tyrosine	3.89	4.43
Phenylalanine	4.98	5.17
Ammonia	2.23	2.05
Recovery, Kjeldahl protein basis, %	101.02	99.79

except in the cases of arginine and methionine. The data were expressed as grams amino acid per 100 grams of protein. The actual recovery was 101.02% and 99.79%, respectively, from these two samples.

SUMMARY AND DISCUSSION

Mature shelled corn was reduced in kernel moisture content from 15.1% to a safe storage value of about 10 to 12% by heated air under forced draft at temperatures of 120°F. (as control), 140°F. and 180°F. (for heat damage).

Samples of whole corn meal, horny endosperm and floury endosperm from the corn dried at different temperatures were used as raw materials from which to extract the protein by the procedure of Osborne and Mendel (29, 54) as modified by Nagy et al. (32) and by Skoch (55). The samples were subjected to successive extraction with each of the following solvents: distilled water, 5% sodium chloride, 80% ethanol plus 0.2% sodium acetate and 0.2% sodium hydroxide.

The protein from corn dried at different temperatures showed qualitatively that some changes in the physical state of the protein took place at drying temperature somewhere above 120°F.

In the whole corn meal, the albumin fraction and the total soluble protein were significantly different after heat treatment. As the temperature at which the corn was dried increased the albumin fraction increased in amount but the total soluble protein decreased. The LSD test gave a significant difference for the control and the heat damaged corn.

The albumin, globulin, prolamine and total soluble protein of the horny endosperm were significantly different after heat treatment. As the temperatures at which the corn was dried increased the protein solubility decreased. The LSD test showed the fractions from corn dried at 120°F. were significantly different from those from corn dried at 140°F. and 180°F. But between 140°F. and 180°F. there was no significant difference resulting from heat treatment. For albumin and globulin fractions there was a significant difference between 120°F. and 180°F. but not between 120°F. and 140°F. or 140°F. and 180°F.

For the floury endosperm the albumin, prolamine and glutelin were significantly different in amounts after the corn had been subjected to different drying temperatures. The albumin and prolamine fractions decreased as the drying temperatures increased but the glutelin fraction increased as the temperature increased. The LSD test for these three fractions showed the temperature of 120°F. had a significant difference from both 140°F. and 180°F. Between 140°F. and 180°F. there was no significant difference for heat treatment.

The amino acid analysis of the total soluble protein from the whole corn meal from corn dried at 120°F. (control) and from that dried at 180°F. (heat damaged) showed no significant difference. Even though the heat damaged corn had 10% less soluble protein than the control, the amino acid composition was the same.

Suggestions for Future Research

The objective of this study was to determine the protein fractions of corn dried at different temperatures. The results

obtained suggested some additional areas for future research.

Amino acid analysis of each protein fraction extracted from samples of whole corn meal, horny endosperm and floury endosperm from corn dried at different temperatures should be made to obtain further insight into the nature of the changes in corn protein when corn is dried artificially. Especial attention is deserved by those fractions which differed significantly after heat treatment. Also by rat feeding experiments the nutritive value of these samples could be evaluated.

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EFFECT OF DRYING TEMPERATURE ON SOLUBILITY OF CORN PROTEIN

by

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AN ABSTRACT OF A MASTER'S THESIS

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KANSAS STATE UNIVERSITY Manhattan, Kansas 1970 Corn harvested at 20 to 30% moisture content by picker-sheller should be artificially dried to 12 to 13% moisture content for safe storage. But too high temperatures often result in overdrying and heat damage which decreases the value of the grain to dry and wet corn millers, cattle feeders and distillers as has been reported by various previous workers.

For the present study, the nature of the changes brought about in corn protein as indicated when fractionated by a modified Osborne-Mendel method was investigated. Samples of whole corn meal, horny endosperm and floury endosperm were subjected to successive extractions with distilled water, 5% sodium chloride solution, 80% ethanol plus 0.2% sodium acetate solution, and 0.2% sodium hydroxide solution. The amounts of these four fractions were added to obtain the value for total soluble proteins.

In these experiments, the corn was dried under relatively mild conditions from an initial moisture content of 15% to 12% or 10% moisture content at temperatures of 120°F., 140°F., and 180°F. Only 3% to 5% moisture was driven out by the heat treatment and therefore the corn was probably not damaged as much as when more drastically dried from 25% to 12% moisture content by some previous workers. However, results showed that total protein solubility decreased as the drying temperature increased from 120°F. to 180°F. Therefore, drying corn at temperatures above 120°F. would be objectionable

from the stand point of reduced protein solubility.

In the whole corn meal, as the temperature at which the corn was dried increased, the albumin fraction increased in amount but the total soluble protein decreased. The solubility of albumin, globulin, prolamine and total soluble protein of the horny endosperm decreased as the temperature at which the corn was dried increased. For the floury endosperm the albumin and prolamine fractions decreased as the drying temperature increased but the glutelin fraction increased as the drying temperature increased.

Except in the case of arginine and methionine, the amino acid analysis of the total soluble protein from the whole corn meal showed no significant difference between the control (corn dried at 120°F.) and the most severely heat-treated (corn dried at 180°F.). The suggestion was made that future work should include the determination of the amino acid content of each extracted fraction.