

Milling Evaluation of Hard Red Spring Wheats. V. Relation of Wheat Protein, Wheat Ash, Bran Pentose, Flour Pentose, and Starch on Bran to Milling Results¹

W. C. SHUEY and K. A. GILLES², North Dakota State University, Fargo 58102

ABSTRACT

Seventeen predominately spring wheat commercial mill mixes were ground on commercial mills, a Pilot mill, a Buhler mill, and a Quadrumat Jr. mill to determine the relation of certain chemical constituents with the milling results. The wheat ash and protein contents were significantly correlated to total flour extraction (0.601* and 0.532*, respectively). Percent starch on bran was highly significantly correlated to flour yield (-0.708**). The pentose-rich fraction from the bran was significantly correlated to the extractions at 0.46% ash for the pilot mill, Buhler mill, and commercial mills (0.566*, 0.523*, and 0.513*, respectively). The pentose-rich fraction from the flour was not significantly correlated with any of the factors studied. Wheat ash, wheat protein, and percent starch on bran are related to the milling characteristics of wheat.

Investigation of chemical constituents of a wheat in relation to milling characteristics and results would appear to reveal useful information for the evaluation of wheat samples. Shuey and co-workers (1,2,3,4) reported on some of the physical characteristics of wheats and milling results in relation to evaluating the milling quality of wheat. Shellenberger and Ward (5) suggested that unless milling is done in accord with full knowledge of the structure and composition of the wheat kernel, the task becomes most difficult. MacMasters et al. (6) reviewed the composition of the wheat kernel and related mill products which varied from year to year and from area to area.

Sharp (7) showed that kernel density is directly related to protein content. The more vitreous kernels within a variety, the higher the protein content. Peters and Katz (8), by a gradient density technique, obtained a range in density of 1.255 to 1.416 g. per cc. for wheat. Baker and co-workers (9) found a correlation coefficient of 0.748 between kernel density and flour yield for hard red winter wheat but a -0.530 coefficient for hard red spring (HRS) wheat and nonsignificant coefficients for the soft red winter and white wheats.

Sherwood and Bailey (10) obtained a correlation of 0.81 ± 0.02 between wheat ash and straight-grade flour ash for 148 samples over three crop years. Zwingelberg (11) stated that wheat ash was dependent on variety, environment, and fertilizer. He found a 0.717 correlation coefficient between wheat ash and flour ash.

¹Cooperative investigation, Plant Science Research Division, Agricultural Research Service, U.S. Department of Agriculture, and the Department of Cereal Chemistry and Technology, North Dakota State University, Fargo. Published with the approval of the Director of the Agricultural Experiment Station, North Dakota State University, Fargo, as Journal Series No. 358. Taken in part from a Ph.D. thesis submitted by W. C. Shuey to the North Dakota State University.

Mention of a trademark name or proprietary product does not constitute a guarantee or warranty of the product by the U.S. Department of Agriculture, and does not imply its approval to the exclusion of other products that may also be suitable.

²Respectively: Research Technologist, Plant Science Research Division, Agricultural Research Service, U.S. Department of Agriculture; and Professor and Vice President for Agriculture, North Dakota State University.

Elder and co-workers (12) found correlation coefficients of -0.70 and -0.75 between pentosans (extracted from ground wheat with 2N hydrochloric acid) and milling score and flour yield, respectively. Weswig et al. (13) obtained correlation coefficients between pentosan values and milling scores of -0.75 and -0.84 for selected winter and spring wheats, respectively. The correlation coefficients between pentosan and flour yield, bran weight, and bran cleanup were significant at

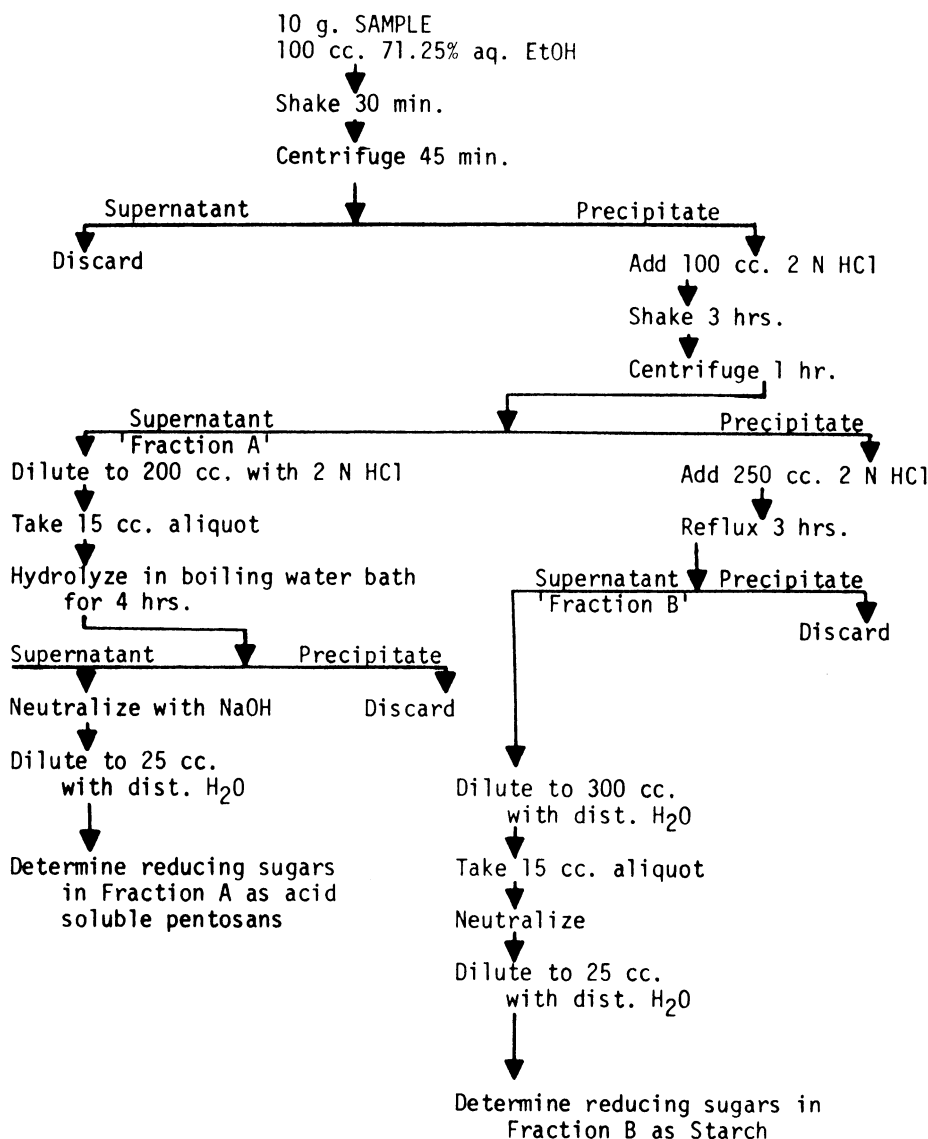


Fig. 1. Extraction scheme for isolation of carbohydrates with hot and cold acid.

the 1% level. However, Baker and co-workers (9) concluded from their study that although pentosan content showed some relation to flour yield for hard red winter and white wheats, the pentosan determination would not be satisfactory for predicting flour yield.

This study was made to determine if certain chemical constituents found in mill products could be related to milling quality.

MATERIALS AND METHODS

Four milling values were developed (1) to determine the dollar value of a 100-lb. wheat sample when converted to 100 lb. of product, namely,

1. Regular or 4-grade operation — a patent flour, 1st clear flour, 2nd clear flour, and feed are produced.
2. Straight-grade or 3-grade operation — a straight-grade flour (usually 0.46% ash), clear flour, and feed are produced.
3. Fancy clear operation — as much fancy clear flour (usually 0.70% ash), patent flour, and feed are produced.
4. The weighted dollar value — based on the average annual industry wide production of the three previous operations.

A value of \$5.80 for patent (0.40% ash), \$5.70 for straight-grade (0.46% ash), \$5.25 for fancy clear (0.70% ash), \$5.00 for 1st clear (0.735% ash), \$3.80 for 2nd clear (1.20% ash), and \$1.70 for feed per 100 lb. was assigned each product for calculating the different dollar values. The weighted dollar value was used in these studies.

The mills employed were:

1. The Brabender Quadrumat Jr. mill, which was the smallest unit capable of grinding 100 lb. per day;
2. the Buhler Pneumatic Continuous mill (500 lb. per day);
3. the Pilot mill (7,200 lb. per day); and
4. ten commercial mills; the largest commercial milling units are capable of grinding over 1,500,000 lb. of wheat per day.

Details of the milling procedure and results were previously given by Shuey et al. (2).

Seventeen 100% HRS or predominately HRS, clean, dry commercial mill mixes were obtained from eight milling companies. Each company furnished 200 lb. each of the wheat mixes representing different protein levels ranging from 12.7 to 16.2%. The clean, dry wheat mixes were representative of the mill run for which the commercial milling performance figures were also obtained.

All analyses were reported on a 14% moisture basis.

The analytical procedures were basically those in the AACC Approved Methods (14).

The flour and bran were extracted as outlined in Fig. 1.

Pentosan-Rich Fraction

The pentosan-rich fractions (fraction A, Fig. 1) contained those sugars which

were extracted by 2N hydrochloric acid (HCl) after the sample had been extracted with 71.25% aqueous ethanol to remove the simple free sugars. The sugars were determined from a mixture of 1 cc. each of nine extracts for both the flour and bran and were de-ionized by shaking the mixture for 15 min. with 0.5 g. each of ion exchange resins, IR 120 (cation) and IR 45 (anion). The mixtures were filtered and concentrated to approximately 1 cc. The concentrated mixtures were spotted on a paper chromatogram and eluted for 10 hr. with a 10:4:3 (v./v./v.) solution ratio of ethyl acetate:pyridine:water and visualized with silver nitrate. The paper chromatogram revealed a small amount of glucose in both the bran and flour extracts with less in the latter. However, the predominate sugar was xylose with approximately the same amount of arabinose in the flour extract and a smaller amount in the bran extract. The chromatogram data and protein content of the extract (less than 1%) from these fractions verified that the sugars are predominately pentoses.

The percentage of sugars was determined by the ferricyanide reducing sugar method (14) using 0.05N ferricyanide. The cubic centimeters of ferricyanide reduced were expressed in milligrams of maltose equivalents and converted to percent pentose by the formula: For bran extract: $\text{mg. maltose} \times 0.8333 = \%$; and for flour extract: $\text{mg. maltose} \times 0.3472 = \%$.

Seven separate samples from the same lot of bran were extracted to determine the standard deviation of the test, which was found to be 0.19%.

Starch on Bran

The residue of extracted bran or flour was refluxed for 4 hr. with 2N HCl to determine the percent starch on bran. The fraction (fraction B, Fig. 1) was neutralized and the amount of reducing sugars determined by the ferricyanide method (14). As was the case for fraction A, the data were expressed in milligrams of maltose and converted to percent starch on bran using the formula: $\% \text{ starch on bran} = \text{mg. maltose} \times 0.7954$. The formula contained a factor to correlate previous results obtained by the acid hydrolysis-enzymatic method.

Likewise, the same samples of bran used for fraction A were analyzed to determine the standard deviation of the test for fraction B; this was established as 0.14%.

RESULTS AND DISCUSSION

To evaluate spring wheats in relation to their milling data (Pilot mill flour extraction at 0.40 and 0.46% ash, Buhler mill flour extraction at 0.46% ash, commercial mill flour extraction at 0.46% ash, percent total Pilot mill flour extraction, and percent total tail shorts from Pilot mill), five wheat or wheat product constituents (wheat protein, wheat ash, pentose-rich fraction of bran, pentose-rich fraction of flour, and starch on bran) were used. The factors studied and the data obtained for the 17 commercial mill mixes are given in Table I. In Table II are the significant correlation coefficients found between the chemical components or constituents and the milling values.

In an earlier paper (4) the weighted dollar value, as well as other calculated comparative milling values (dollar value for 4-grade operation, straight-grade operation, and fancy clear operation; milling rate at 0.40 and 0.46% ash; ash value; curve index; and inverse curve index), was discussed which is related to the

TABLE I. CHEMICAL DATA ON COMMERCIAL WHEAT MIXES

Mix	Wheat		Pentose-Rich Fraction		Starch on Bran %
	Protein ^a %	Ash ^a %	Bran %	Flour %	
1A	12.9	1.507	2.17	0.97	5.33
2A	15.2	1.650	2.92	0.83	7.28
1B	13.2	1.613	2.42	0.97	8.11
2B	15.2	1.610	2.67	0.83	6.12
1C	13.6	1.602	3.00	1.01	7.30
2C	15.3	1.698	3.08	0.94	7.08
1D	15.3	1.600	3.16	1.09	7.13
2D	16.2	1.695	3.13	0.96	7.78
1E	14.1	1.564	2.67	0.87	6.92
2E	14.4	1.614	2.81	1.10	7.51
1F	13.3	1.573	2.83	0.63	7.00
2F	15.2	1.563	2.33	0.83	6.68
1G	14.6	1.512	2.50	0.90	6.72
2G	14.7	1.627	2.42	0.89	6.20
1H	13.5	1.590	2.67	0.96	6.36
2H	14.9	1.581	2.92	0.92	6.84
3H	12.7	1.515	2.75	0.85	6.48

^a14% Moisture basis.TABLE II. SIGNIFICANT CORRELATION COEFFICIENTS^a BETWEEN CHEMICAL CONSTITUENTS AND MILLING DATA

Constituent	P.M. Ext. ^b		B.M. Ext. ^b at 0.46	C.M. Ext. ^b at 0.46	Percent Total Flour ^c	Percent Tail Shorts ^c
	at 0.40	at 0.46				
Protein	N.S.	N.S.	-0.548*	N.S.	-0.601*	0.563*
Ash	N.S.	-0.707**	-0.710**	N.S.	-0.521*	N.S.
Bran pentoses	N.S.	-0.566*	-0.523*	-0.513*	N.S.	N.S.
Starch on bran	-0.523*	-0.592*	-0.572*	N.S.	-0.708**	N.S.

^aN.S. = not significant; * = significant at 5% level; ** = highly significant at 1% level.^bP.M. = Pilot mill; B.M. = Buhler mill; C. M. = commercial mill.^cFor the Pilot mill.

economic worth of a wheat and indirectly associated to the milling quality. It was found that the weighted dollar value correlated most consistently with the milling data at the highly significant level of 1% and with total Pilot mill flour extraction gave a correlation coefficient of 0.635**. Two wheat physical characteristics, test weight and 1,000-kernel weight, also gave highly significant correlation coefficients with total Pilot mill flour extraction, 0.716** and 0.650**, respectively (3). Of the five constituents studied, only percent starch on bran was found to be highly significantly correlated with total Pilot mill flour extraction (0.708**). Because of the milling technique employed, the total Pilot mill flour extraction is an indicator of the ease of the "millability" of the wheat sample, thus a high extraction should indicate an easy milling wheat. It was surprising that there was not a significant correlation between percent bran and percent starch on bran. These four values (test weight, 1,000-kernel weight, percent starch on bran, and weighted dollar

TABLE III. CORRELATION COEFFICIENT^a MATRIX OF TEST WEIGHT, 1,000-KERNEL WEIGHT, PERCENT STARCH ON BRAN, AND DOLLAR VALUE

	Test Weight	1,000-Kernel Weight	Percent Starch on Bran	Dollar Value
Test weight	...	0.744**	N.S.	0.537*
1,000-Kernel weight		...	N.S.	0.560*
Percent starch on bran			...	-0.720**
Dollar value				...

^aN.S. = not significant; * = significant at 5% level; ** = highly significant at 1% level.

value) were significantly correlated among themselves, with the exception of percent starch on bran with test weight and 1,000-kernel weight as given in Table III. There is no explanation as to why test weight or 1,000-kernel weight is not correlated to starch on bran unless that density, size, and shape of the kernel are not related to the ease of separation of the bran and the endosperm, but do influence the total flour obtained. This may be particularly true for density or compactness of the kernel's outer portion, since no significant correlation even at the 5% level was found between percent starch on bran and percent bran.

Besides starch on bran, protein and ash were correlated to the total flour extraction (-0.601* and -0.521*, respectively). All three are thought to be related in some manner directly or indirectly to the texture of the kernel and protein — the most closely associated with vitreousness. Wheat protein content was negatively and highly significantly correlated to test weight and 1,000-kernel weight (-0.714** and -0.652**, respectively), whereas wheat ash was only significantly negatively correlated to test weight (-0.482*). Percent starch on bran was not significantly correlated to wheat protein content, but was significantly correlated to wheat ash (0.550*).

The bran pentose-rich fraction and the starch on bran were negatively correlated to the dollar value (-0.516* and -0.720**, respectively), and milling ratings at 0.40 and 0.46% ash (-0.598*, -0.551*, and -0.761**, -0.754**, respectively), and positively correlated to the ash value (0.495* and 0.677**, respectively). The percent extraction as related to flour ash was involved in the calculation of these values (dollar value, milling ratings, and ash values), yet the bran pentose-rich fraction was not significantly correlated to the total Pilot mill flour extraction. Thus, it would appear that bran pentoses are associated with the relative friability and tenacity between sections of the bran and cells in the immediate proximity.

The flour pentose-rich fraction was not significantly correlated with any of the factors studied.

CONCLUSIONS

Wheat ash and protein contents were significantly correlated to total flour extraction, and percent starch on bran was highly significantly correlated to flour yield. This would indicate that wheat ash, wheat protein, and percent starch on bran are related to the milling characteristics of the wheat. The bran pentose-rich fraction was significantly correlated to the extractions at 0.46% ash for the Pilot mill, Buhler mill, and commercial mills, as well as to the calculated values for dollar

value, milling ratings, and ash value. Because of the interdependence of these values to extraction, the pentoses may play a role in the friability of the wheat. The flour pentose-rich fraction was not significantly correlated with any factors studied.

There are two distinct and apparently independent broad classes of factors which influence total Pilot mill flour extraction: 1) Physical characteristics – size, shape, density, etc., and 2) chemical components – protein, ash, starch, pentosans, etc.

The inter- and intrarelations of these two classes are complex and ill defined.

Acknowledgments

The authors wish to thank R. D. Crawford, R. D. Maneval, and K. J. Sprick for their assistance in this study.

Literature Cited

1. SHUEY, W. C., SPRICK, K. J., and GILLES, K. A. Milling evaluation of hard red spring wheats. Part I. Dollar value. *Northwest. Miller* 278: 9 (1971).
2. SHUEY, W. C., GILLES, K. A., and MANEVAL, R. D. Milling evaluation of hard red spring wheats. Part II. Comparison of mills and milling results. *Northwest. Miller* 278(11): 12; (12): 11 (1971).
3. SHUEY, W. C., and GILLES, K. A. Milling evaluation of hard red spring wheats. Part III. Relation of some physical characteristics to milling. *Northwest. Miller* 279(2): 14 (1972).
4. SHUEY, W. C., and GILLES, K. A. Milling evaluation of hard red spring wheats. Part IV. Relation of calculated milling values to milling results. *Northwest. Miller* 279(3): 14 (1972).
5. SHELLENBERGER, J. A., and WARD, A. B. Experimental milling. In: *Wheat and wheat improvement*, Chapt. 13. American Society of Agronomy: Madison, Wisc. (1967).
6. MacMASTERS, M. M., HINTON, J. J. C., and BRADBURY, D. Microscopic structure and composition of the wheat kernel. In: *Wheat: Chemistry and technology*, ed. by Y. Pomeranz (Rev.). Amer. Ass. Cereal Chemists: St. Paul, Minn. (1971).
7. SHARP, P. F. Wheat and flour studies. IX. Density of wheat as influenced by freezing, stage of development, and moisture content. *Cereal Chem.* 4: 14 (1927).
8. PETERS, W. R., and KATZ, R. Using a density gradient column to determine wheat density. *Cereal Chem.* 39: 487 (1962).
9. BAKER, D., FIFIELD, C. C., and HARTSING, T. F. Factors related to the flour yielding capacity of wheat. *Northwest. Miller* 272: 16 (1965).
10. SHERWOOD, R. C., and BAILEY, C. H. Correlation of ash content of wheat and flour. *Cereal Chem.* 5: 437 (1928).
11. ZWINGELBERG, H. Beziehungen Zwischen Ganzkornaschegehalt, Mehlaschegehalt, und Mehlfarbe bei Weizensorten. *Getreide Mehl* 10: 117 (1961).
12. ELDER, A. H., LUBISICH, T. M., and MECHAM, D. K. Studies on the relation of pentosans extracted by mild acid treatments to milling properties of Pacific Northwest wheat varieties. *Cereal Chem.* 30: 103 (1953).
13. WESWIG, P. H., FOOTE, W. H., and DRUSTS, G. R. The acid-extracted pentosan content of wheat as a measure of milling quality of Pacific Northwest wheats. *Cereal Chem.* 40: 169 (1963).
14. AMERICAN ASSOCIATION OF CEREAL CHEMISTS. AACC Approved methods (7th ed.). The Association: St. Paul, Minn. (1962).

[Received November 29, 1971. Accepted August 8, 1972]