CEREAL CHEMISTRY

VOL. 37 SEPTEMBER, 1960 NO. 5

THE SUBSIEVE-SIZE FRACTIONS OF A HARD RED SPRING WHEAT FLOUR PRODUCED BY AIR CLASSIFICATION[1]

REZSOE GRACZA[2]

ABSTRACT

A conventionally milled hard red spring wheat flour was fractionated by repeated air classifications.

The repetitive procedure, starting with separation at low particle size, produced fractions with protein contents as high as 20.3% and as low as 8.8% from a parent flour of 13.8% protein content and 86% extraction (flour basis) of a 100% Montana hard spring wheat.

The degree of protein shifting index shows that 5.4% of the parent flour's 13.8% protein could be shifted within the fractions, compared to 27.7% protein shifting in the identically processed 7.8% protein content soft red winter wheat flour described in a previous paper.

Other indices such as ash, diastatic activity, alkaline water retention capacity, thiamine, fat, and MacMichael viscosity appreciably increased with increasing protein content and/or specific surface within the subsieve size range. Bulk density increased with increasing particle size, whereas specific gravity decreased with increasing protein content in the fractions. The pH value reached its maximum in the fraction of lowest protein content.

Physical dough test data indicate that the dough development time increased with increasing protein content. Absorption, however, increased not only with the protein content but also with decreasing particle size, i.e., increasing specific surface. Doughs made from flour particles collected in the 44.5 to 48.0 SED micron size range yielded extensigrams characteristic of good bread flours, whereas doughs made from flour particles collected in the 23.0 to 39.0 SED micron size range produced farinograms and extensigrams characteristic of soft wheat flours used in pastries.

This paper reports the response of a hard red spring wheat flour to a repeated air-classification procedure described in a previous paper (8) which showed the response of a soft red winter wheat flour to the same treatment. The use of identical procedures and about the same analytical indices facilitates a comparison of the behavior of hard and soft wheat flours in air classification.

In the former paper (8), the literature review was restricted to size

[1] Manuscript received October 3, 1959.

[2] Address: The Pillsbury Company, Minneapolis, Minnesota.

reduction and size classification studies closely related to the subject matter of the paper. Since, in a broader sense, histological observations and protein isolation from flour are related to the subject matter, some extension of the references of the previous article is here offered.

Histological studies of the wheat, and especially of the endosperm, are probably as old as the microscope itself. At the turn of the century, Cobb (6) formulated the obvious observation that "the starch granules in each flour-cell of the ripe (wheat) grain are held enmeshed in the dry and tough elaborate network of protoplasmic matter which, when soaked, assembled, and weighed by the chemist, is entered in his returns under the name of gluten." He further stated that "the tendency of the starch grains (granules) during the process of making flour is to fly loose." On the basis of this observation, Cobb was able to shift some protein in a flour obtained from the innermost section of the wheat kernel, using a 112-mesh "fine silk" sieve. He is probably the first to record protein shifting by recognizing the heterogeneity of the wheat endosperm. Greer *et al.* (9) elaborated, in their morphological studies, on the different shapes and sizes of the wheat endosperm fragments with particular reference to the locational origin of fragments and cell walls attached to them. This morphological study was continued by Kent and Jones (14) with particular reference to distinguishing between the endosperm fragments of break and reduction flours.

Hess and Hanssen (13) described a procedure for the production of native vegetable protein (and cereal protein) using density separation in nonaqueous liquids advantageously selected to float the protein material while the heavier constituents of the flour could be separately collected. Their work was aimed at producing protein in a native form, as contrasted to the commercially washed gluten obtained by methods first described by Beccari (5). Studying flour and protein matrix obtained by density separation, Hess observed that individual starch granules and some protein particles were freed during milling (11). Hanssen *et al.* (10) measured the size distribution of different cereal and leguminous starches using a microscope. They found that starches in the wheat flour range in diameter from 0.55 to 55 microns and the "protein lamellae" obtained by density separation have a thickness of 0.26 micron. The latter measurement was based on the average surface area of protein particles (50 square microns) as measured by microscopic observation.

These studies reveal that the concentration or isolation of proteinaceous substances from cereal flours may be obtained by different means such as 1) by washing out the starch granules with the use of aqueous media (5); 2) by flotation in nonaqueous liquid media using density differences between the protein and the starch granules (13); and 3) by flowdynamic classification utilizing simultaneously the size, density, and shape of the partly homogeneous and partly heterogeneous flour particles (8).

Applying different principles, different degrees of protein concentration and/or isolation are obtained. Owing to the different principles involved, the protein and starch products obtained by the different methods exhibit differences. Such differences were recorded in detail by Hess (12) in comparing water-washed gluten with protein material obtained by density separation.

Materials and Methods

The parent flour was an unbleached, untreated 86% extraction (flour basis) patent flour milled commercially from 100% Montana hard red spring wheat having 16% protein content and 60.0 lb. per bushel test weight measured at 12.1% moisture. This parent flour was fractionated, without additional grinding, 4 months later. Thus, the separations can be considered typical of what may be obtained from spring wheat flours by this classification procedure.

This parent flour was fractionated with process variables identical to those used for the soft wheat flour fractionation (8) in a commercial-size air-classifier which was provided with a special, forced vortex type rotor in the classification zone similar to the type described by Lykken (15). This fractionation produced seven subsieve-size fractions from the parent hard wheat flour.

The flowsheet (Fig. 1) identifies the samples with capital letters and indicates the critical-cut size of the classification obtained by analysis of the particle size distribution curves of the fine and coarse flour fractions described in a previous paper (8). The parent stock was subjected to a first-stage air separation at 21 SED micron critical cut resulting in a fine fraction, A, and in a coarse fraction, AA. The first-stage coarse fraction, AA, was subsequently subjected to a second-stage air separation at a larger critical cut (23 SED micron), resulting in a fine fraction, B, and in a coarse fraction, BB. The classification of the coarse fractions was repeated five times, so that the total procedure resulted in seven fractions, namely A, B, C, D, E, F, and FF.

The description of the critical cut and sharpness figures and their interpretations are described in the previous paper (8). The method used to obtain the particle size data was a slight modification of the method described by Whitby (16).

The average particle diameter figures were obtained by the Fisher

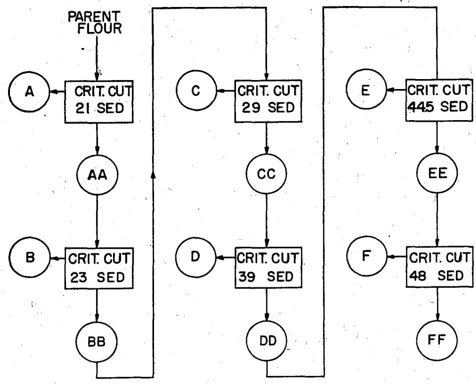

Fig. 1. Flow diagram of the repetitive classification procedure with increasing critical-cut sizes.

subsieve-sizer apparatus (7) which uses the principle of permeability through a porous bed.

The following analyses were made with slight modifications of the procedures described in *Cereal Laboratory Methods* and identified by their test numbers (1): protein 67.1, moisture 48.3, ash 9.1, diastatic activity 24.1, pH 60.2, thiamine 98.8c, and MacMichael viscosity 97.1. Specific gravity was determined by ASTM Method C-188 (3), petroleum ether-extractable fat by AOAC Method 22.26 (4), and free fatty acid content by AOCS Method CA5a-40 (2). The two last-mentioned methods were modified slightly. Alkaline water retention capacity was obtained by the method of Yamazaki (17). Color reflectance values were obtained from a kerosene-flour slurry measured on glass with a Gardner Differential Tristimulus colorimeter. Bulk density data were obtained by the method described in the previous paper (8).

Farinograms were obtained using the 300-g. mixer and the Constant Flour Method (Method 26.4). The extensigrams were determined by the procedure described previously (8), except that all samples were tested by the longer remix option and the 3-hour schedule normally used for bread flours. The scale resistance was set at 2:1; i.e., 1,000 g. resistance registers 500 B.u. Resistance to extensibility was measured at 500 mm. deformation uncorrected for cradle travel.

Results and Discussion

Microscopic Studies. Figures 2 and 3 are photomicrographs of the hard spring wheat parent flour and its seven subsieve-size fractions using 180× magnification.

Comparison with the photomicrographs of the soft wheat flour fractions (8) reveals the same trend in the proportion of protein matter to starch granules that was observed in the soft wheat flour fractions. The following differences are evident:

a. The elementary protein matter particles appear to be smaller, thinner, and less irregular in shape than those from the soft wheat flour. This is evident in the A and B fractions.
b. The elementary starch granule particles generally appear to have a flatter lenticular shape than those from soft wheat flour. These flatter granules split into halves or into sectors of an ovoid shape

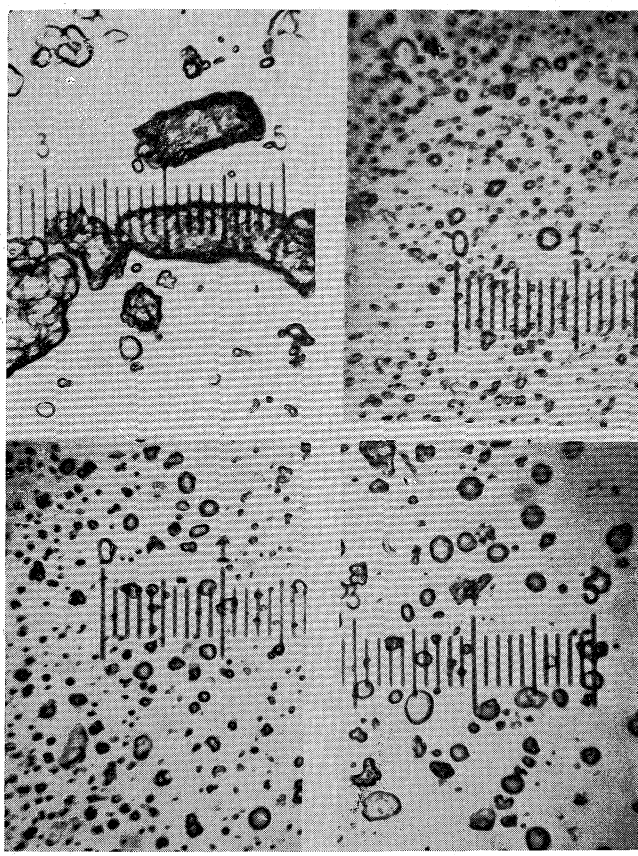

Fig. 2. Photomicrographs of the parent flour (upper left), the A fraction (upper right), the B fraction (lower left), and the C fraction (lower right). All photographs were taken at 180× magnification. Slides prepared in an oil of 1.600 refractive index.

more frequently than those from a soft wheat flour as seen in the C, D, and E fractions.

c. The surfaces of the individual starch granules or starch granules in smaller aggregates do not appear to have been stripped from the protein matrix portions as cleanly as those originating from soft wheat flour. This is evident in all fractions to different degrees except the last fraction (FF).

d. The number of large elementary starch granules with or without some protein matrix on their surface is considerably smaller than that observed in a soft wheat flour; note the photograph of the parent stock.

e. Endosperm chunk particles have polygonal shapes with definite edges, a large portion of the cells being similar in shape to a par-

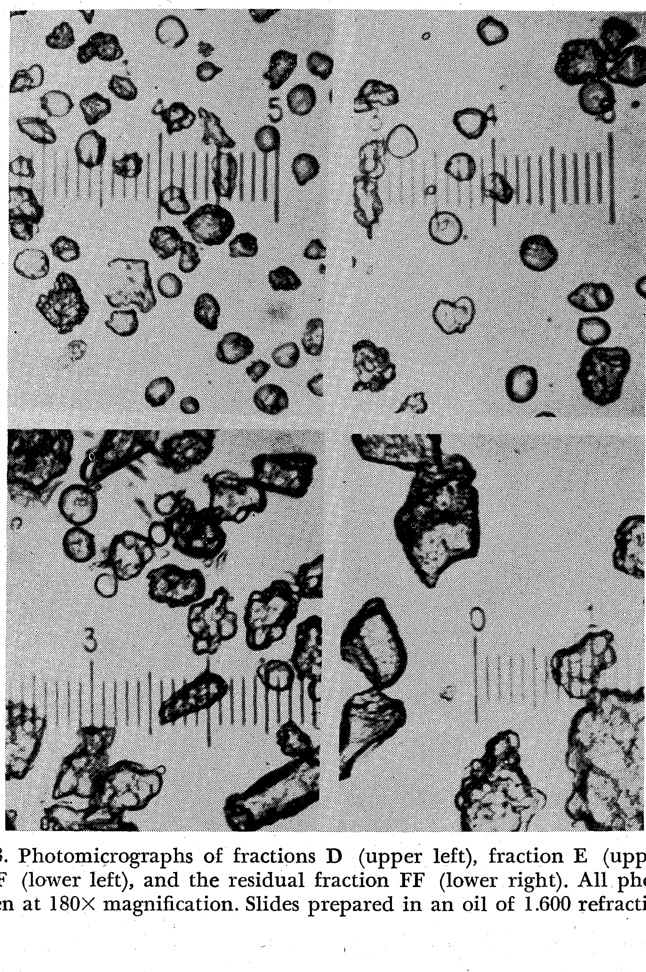

Fig. 3. Photomicrographs of fractions D (upper left), fraction E (upper right), fraction F (lower left), and the residual fraction FF (lower right). All photographs were taken at 180× magnification. Slides prepared in an oil of 1.600 refractive index.

allelepiped. Soft wheat flour endosperm chunk particles, in comparison, have a rounded-off shape and smeared-off, furry edges with starch granules occasionally protruding from the body of the particle. See parent and FF fractions.

f. Many of the endosperm chunk particles contain large fissures, a bodily discontinuation which is practically absent from chunk particles of soft wheat origin.

Protein Content of the Fractions. The protein histogram, Fig. 4, illustrates that the classification steps resulted in a concentration of

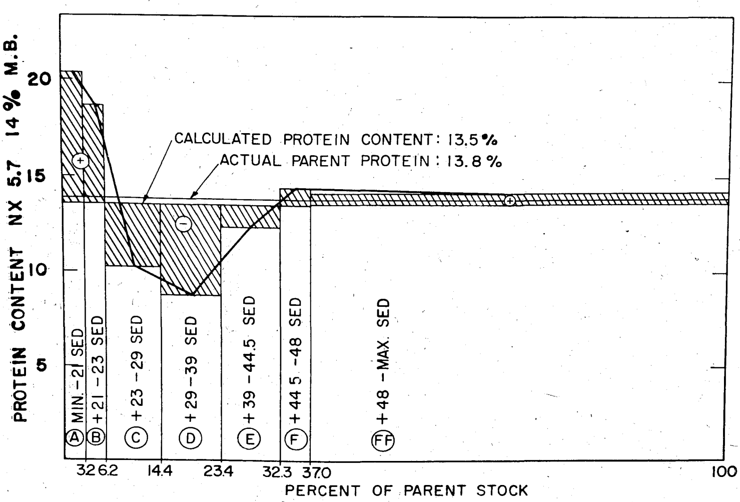

Fig. 4. Histogram of protein content versus percent of parent stock from the seven subsieve-size fractions of a hard red spring wheat flour, illustrating the positive and negative protein shifting in the respective fractions.

protein in fractions A, B, F, and FF, while fractions C, D, and E were depleted of protein as compared to the distribution of protein and starch in the parent flour.

Utilizing the term *degree of protein shifting*[3] employed in the previous paper, a comparison can be drawn between the response of the soft red winter wheat flour and that of the hard red spring wheat flour. It was possible to shift 5.4% of the hard wheat flour's protein content within the seven fractions, whereas 27.7% of the soft wheat flour's protein was shifted in an identical classification procedure.

[3] Degree of protein shifting expresses the percentage of the protein content of the original flour which was shifted into or out of the specific fractions as a result of air classification. Of necessity, the portion of protein which was shifted out of the low-protein fractions has to be found in the high-protein fractions (8).

From a fitted curve connecting the midpoints of the bars, or by equating areas under and above the curve, it is possible to estimate yields at a chosen protein content. Another useful form of such a histogram is made by plotting the midpoints of the protein content as a function of particle size. This permits the estimation of the protein content per size interval or the protein content versus particle size if infinitesimally small size intervals are used.

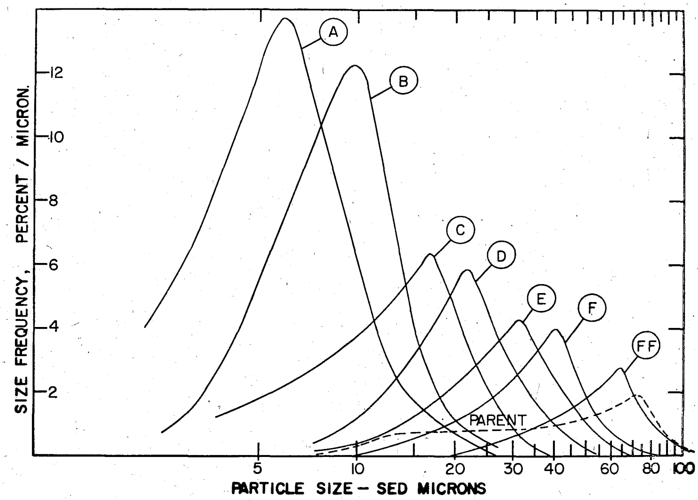

Fig. 5. Size distribution curves of the parent flour and its seven subsieve-size fractions plotted as percent per micron versus logarithm of size.

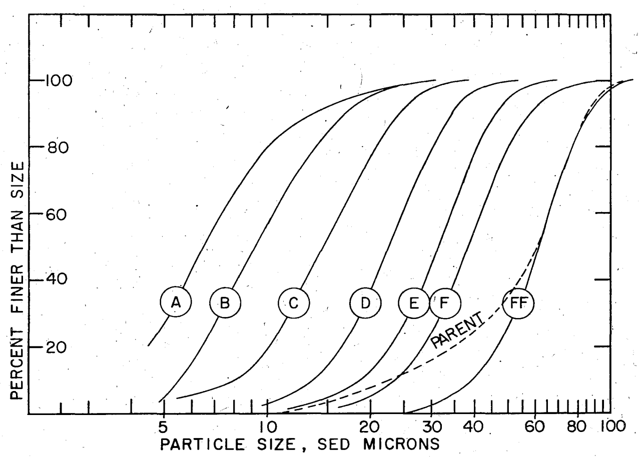

Fig. 6. Cumulative size distribution curves of the parent flour and its seven subsieve-size fractions. Capital letters A, B, C, D, E, F, and FF designate increasingly coarser fractions.

Particle Size Data. The particle size distribution curves of the parent flour and its fractions are presented in Figs. 5 and 6, where the same distribution functions are plotted in percent/micron versus log SED size, and percent finer than size versus log SED size. These curve families are indicative of the closeness of the critical-cut size ranges of the fractions.

Table I presents the numerical data for particle size distribution as obtained by the slightly modified Whitby centrifuge technique (16).

TABLE I

PARTICLE SIZE DATA OF PARENT FLOUR AND ITS SEVEN SUBSIEVE-SIZE FRACTIONS, OBTAINED BY THE MODIFIED WHITBY SEDIMENTATION METHOD

SED Micron	Percent Finer than Size of the Parent Flour and Fractions							
	Parent	A[a]	B	C	D	E	F	FF
120	100.0	....	....	....	....	....	....	100.0
100	98.0	....	....	....	....	....	100.0	97.1
80	85.6	....	....	....	....	100.0	98.8	86.8
60	47.7	....	....	....	100.0	98.9	92.7	46.6
40	23.8	....	100.0	100.0	97.2	79.0	55.1	10.5
30	15.5	100.0	98.7	97.5	79.7	44.2	23.0	2.7
20	7.7	97.2	94.7	75.7	35.3	12.5	6.6	0.0
10	0.5	80.4	55.3	18.0	2.3	0.6	2.4	0.0
5	0.0	27.0	4.0	4.4	0.0	0.0	0.0	0.0

[a] The sequence of the separation is such that the size increases from A through FF. A through F are the fine fractions of the six process steps, FF being the coarse fraction of the last step.

A cumulative curve of the protein content of the fine and coarse fractions respectively, plotted against critical-cut size, is shown on Fig. 7. The two converging curves meet at the neutral critical cut (8) 31.3 SED micron. The curve pair may be useful as it provides an approximation of the protein content of the coarse and fine products if the parent stock is processed at an arbitrary critical cut as shown on the abscissa.

Other Analytical Indices. The results of other analyses carried out on the parent flour and the subsieve-size fractions are recorded in Table II.

The moisture content in the fractions decreased with smaller average particle size (Fisher); a greater moisture loss during air classification would naturally be expected in those fractions which possess high specific surfaces. Ash content is generally higher in the fine fractions and apparently varies with the protein content of the fraction, except in the coarsest fraction, DD.

Diastatic activity values, when compared to a similar separation of soft wheat flour, are considerably higher though they follow a similar pattern. The higher diastatic activity values are evident where cleaved

TABLE II

ANALYTICAL DATA FOR PARENT FLOUR AND SEVEN SUBSIEVE-SIZE FRACTIONS OF A MONTANA HARD SPRING WHEAT FLOUR

Product	Yield as Per-cent of Parent	Critical Cut Size Range, SED Micron	Sharp-ness	Av. Par-ticle Size (Fisher)	Pro-tein[a] N × 5.7	Mois-ture	Ash	Diastatic Activity, mg. Maltose per 10 g.	AWRC[b]	pH	Specific Gravity	Bulk Density	Color Reflect-ance Rd by Hunter	Thia-mine	Vis-cosity	Petroleum Ether-Extract-able Fat	Free Fatty Acid Fat Basis
			%	µ	%	%	%		%		g/cm³	g/cm³		mg/lb	°MacM	%	%
Parent	100.0	min.-max.	..	23.2	13.8	11.4	0.400	258	74.2	5.82	1.447	0.612	53.7	0.39	197	1.06	15.1
A	3.2	min.-21	82	4.3	20.3	8.9	0.742	528	138.0	5.90	1.430	0.263	58.5	1.96	+300	2.09	24.9
B	3.0	21-23	81	5.2	18.7	8.8	0.749	490	130.0	5.92	1.431	0.294	60.0	1.53	219	1.84	21.7
C	8.2	23-29	79	9.6	10.3	9.5	0.504	523	109.0	5.98	1.458	0.440	61.7	0.61	110	1.04	20.2
D	9.0	29-39	76	14.6	8.8	10.8	0.443	379	67.7	6.00	1.465	0.567	60.4	0.48	56	0.89	18.0
E	8.9	39-44.5	71	17.0	12.4	10.9	0.484	282	70.8	6.02	1.458	0.616	55.2	0.48	131	1.04	17.3
F	4.7	44.5-48	61	19.5	14.5	10.9	0.455	213	71.2	5.95	1.451	0.641	52.3	0.48	182	1.10	16.4
FF	63.0	48-max.	..	30.0	14.2	10.7	0.325	129	63.5	5.73	1.450	0.762	53.0	0.22	146	0.97	14.4
AA	96.8	+21-max.	..	24.0	13.3	11.3	0.394	237	68.7								
BB	93.8	+23-max.	..	24.2	13.0	11.0	0.375	214	66.6								
CC	85.6	+29-max.	..	26.2	13.6	10.5	0.350	187	63.3								
DD	76.6	+39-max.	..	28.0	14.2	10.7	0.354	161	63.3								
EE	67.7	+44.5-max.	..	29.8	14.3	10.7	0.333	141	60.5								

[a] Protein and other test results are expressed on a 14% moisture basis.
[b] Alkaline water retention capacity.

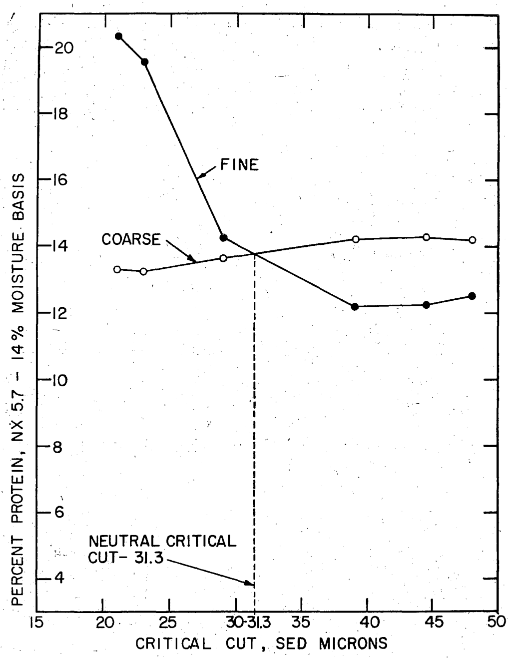

Fig. 7. Protein content of the fine and coarse fractions plotted as functions of critical cut, illustrating the neutral critical cut for a commercial hard spring wheat flour.

starch granules and/or the enzyme accumulate, i.e. in the lower size groups. Alkaline water retention capacity distribution in the fractions, as with soft wheat flour separations, follows primarily the specific surface and/or the protein content and perhaps other components.

The free fatty acid portion of the fats, as shown by the free fatty acid content expressed on a fat basis, increases in the fractions with decreasing particle size.

The specific gravity of the fractions increases with a decrease in protein content, a trend which was also observed in the fractionation of soft wheat flour (8).

Physical Dough Test Data. The farinograms and extensigrams (Figs. 8, 9; Table III) for doughs made from the various fractions show the same general trends observed in doughs made from sofe wheat fractions (8). Dough development times increase with the protein content. Absorption, however, does not increase directly with protein content but varies also with the surface characteristics of the fractions; with higher specific surface, the absorption increases. The major changes in

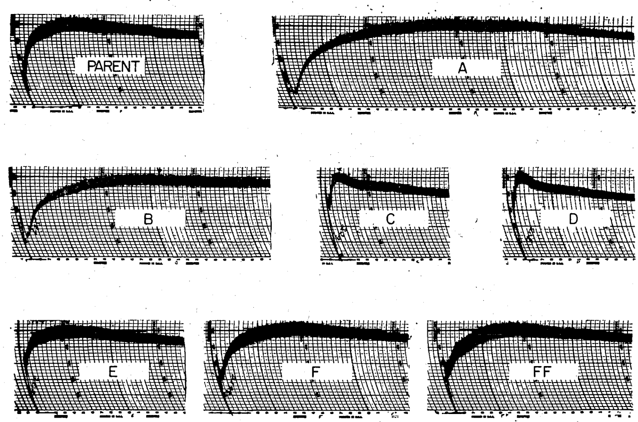

Fig. 8. Farinograph curve characteristics of the parent flour and the seven subsieve-size fractions.

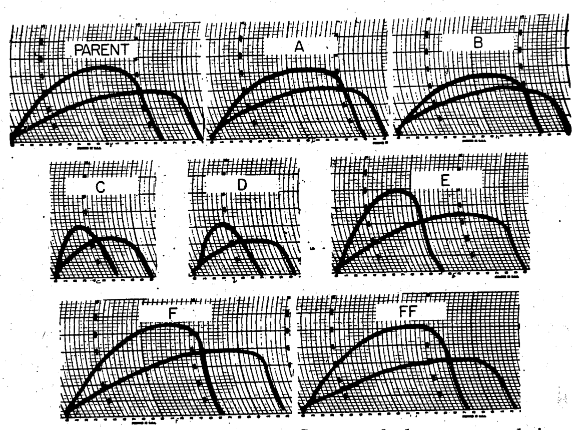

Fig. 9. Extensigraph curves of the parent flour and the seven subsieve-size fractions.

the dough-handling properties are illustrated by the area under the 1-hour extensigraph curves; the area increases about threefold as the protein increases from the lowest to the highest values. This compares to a tenfold change in the fractions of a soft wheat flour (8), indicating also the poorer protein-shifting possibilities in hard spring wheat flours. The arrival time plus dough development time in the finest fractions (A, B) of the hard wheat flour is about half as long as that in the soft wheat flour, indicating an increased rate of hydration at approximately a comparative protein level and a somewhat higher absorption.

While the highest extensibility and/or resistance desired for bread-baking is exhibited in the finest fractions of a soft wheat flour

TABLE III

Major Indices from the Farinograph and Extensigraph Tests of a Hard Red Spring Wheat Flour and Its Seven Subsieve-Size Fractions

Product	Absorption	Farinograph					Extensigraph					
		Dimension[a]				Valorimeter	One Hour			Three Hours		
		A	B	C	D		Extensibility	Resistance	Area	Extensibility	Resistance	Area
	%						*mm*	*B.u.*	*cm*^{2}	*mm*	*B.u.*	*cm*^{2}
Parent	64.7	3.50	6.50	9	30	64	198	155	76	157	285	89
A	89.7	12.50	20	17.25	5	95	181	180	71	160	270	86
B	89.9	8	16	17.50	10	91	181	170	64	152	240	73
C	72.6	1.25	2	1.75	50	41	98	220	32	65	300	23
D	63.7	1.25	1.50	1.25	95	34	105	190	30	70	315	28
E	62.6	2	5.25	8.75	30	59	199	170	86	113	365	66
F	66.9	5.25	9	10.75	30	73	228	170	110	163	320	114
FF	64.4	4.75	9	11.50	30	73	226	145	87	175	280	107

[a] A, time in minutes from the start of water addition until the top of the curve intercepts the 500-B.u. line, "arrival time." B, C, and D, respectively, equivalent to Dough Development Time, Stability, and Tolerance Index (*Cereal Lab. Methods*, 6th ed., sec. 26.4 (see ref. 1)).

from the lower end of the wheat hardness spectrum (8), the same can be observed in the coarsest fractions of hard spring wheat flour from the highest end of the wheat hardness spectrum. The particles collected in the F fraction produced dough development properties optimum for bread-baking. In economic considerations the removal of a portion of the parent flour, such as fractions C and D with poor extensibility, will enhance desired flour properties in bread-baking. The farinograph and extensigraph characters of the C and D fractions are practically identical to those desired in a pastry flour milled from soft wheat.

Thiamine content generally preponderates in the high-protein fractions, though the over-all trend does not follow the protein content but rather the specific surface.

The bulk density data show a general decrease with increasing specific surface.

Soft wheat flour is inherently low in protein content but more responsive to air classification. It is adaptable to the withdrawal of a high-protein fraction leaving fractions applicable to cake flours and low-protein or starchlike applications.

Hard wheat flour, however, shows protein shifting to a lesser degree but the resulting fractions offer a different set of products. Fractions with protein contents and properties may be produced which cover the desirable properties of both pastry- and bread-baking. Thus, a great amount of latitude is available within which properties of flour may be altered by air classification or blending.

Literature Cited

1. American Association of Cereal Chemists. Cereal laboratory methods (6th ed.). The Association: St. Paul, Minnesota (1957).
2. American Oil Chemists' Society. Official and tentative methods (2nd ed.); CA5a-40. The Association: Chicago, Illinois (1946).
3. American Society for Testing Materials. ASTM Standards, 1946, Part II, p. 78 (1946).
4. Association of Official Agricultural Chemists. Official methods of analysis (8th ed.), p. 371. The Association: Washington, D.C. (1955).
5. Beccari. De Bononiensi Scientiarum et Artium Instituto Atque Academia Commentarii. Vol. II, Part 1, p. 122 (1745). (Excerpted by A. Maurizio in: Die Nahrungsmittel aus Getreide, Vol. I, Part 8, p. 404. Paul Parey: Berlin (1917).)
6. Cobb, N. A. Universal nomenclature of wheat. Agr. Gaz. N. S. Wales, Misc. Pub. No. 539, Dept. of Agriculture, New South Wales (1905).
7. Gooden, E. L. Air permeation apparatus for measuring the coarseness of powders. U. S. Patent No. 2,261,802 (November 4, 1941).
8. Gracza, Rezsoe. The subsieve-size fractions of a soft wheat flour. Cereal Chem. **36:** 465–487 (1959).
9. Greer, E. N., Hinton, J. J. C., Jones, C. R., and Kent, N. L. The occurrence of endosperm cells in wheat flour. Cereal Chem. **28:** 58–67 (1951).
10. Hanssen, E., Dodt, Erika, and Niemann, E. G. Bestimmung von Korngrösse Kornoberfläche, und Korngewicht bei pflanzlichen Stärken. Kolloid-Z. **130:** 19–31 (1953).

11. HESS, K. Müllerei und Forschung. Die Mühle **89:** 428–431 (1952).
12. HESS, K. Changes in the protein and starch of wheat flour during mixing and baking. Trans. Am. Assoc. Cereal Chemists **11:** 153–166 (1953).
13. HESS, K., and HANSSEN, E. Verfahren zur Gewinnung von nativem, pflanzlichem Eiweiss. German Patent No. 960,331 (March 21, 1957).
14. KENT, N. L., and JONES, C. R. The cellular structure of wheat flour. Cereal Chem. **29:** 383–398 (1952).
15. LYKKEN, H. G. Centripetal classifier. U. S. Patent 2,741,336 (April 10, 1956).
16. WHITBY, K. T. A rapid general purpose centrifuge sedimentation method for measurement of size distribution of small particles. Part I. Apparatus and method. Heating, Piping and Air Conditioning **27:** 231–237 (January 1955); Part II. Procedures and applications, pp. 139–145 (June 1955).
17. YAMAZAKI, W. T. An alkaline water retention capacity test for the evaluation of cookie baking potentialities of soft winter wheat flours. Cereal Chem. **30:** 242–246 (1953).