Soft Wheat Flour Particle-Size Analysis by Integrated Sieve and Coulter Counter Procedures[1]

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ABSTRACT

A procedure is described for particle-size distribution analysis of coarse soft wheat flour. Sieving was integrated with Coulter Counter analysis of particles at two suspension concentrations. Flours from pure-variety wheats milled under conditions producing varying proportions of coarse, medium, or fine particles gave mass-median diameters and 80%-range values indicative of inherent endosperm texture as reflected by Particle Size Index. Relative granulation characteristics of varieties were not adversely affected by season of crop growth.

Particle-size distribution analysis has been conducted by microscopes, sedimentation, sieving, and similar means. While these procedures are suitable for use with many materials, flour offers some difficulty in analysis because of its shape irregularity, and the density differences among its components. The Coulter Counter[3] method of size analysis, which depends on particle volume only, appears to provide a means of overcoming these difficulties.

Several papers have appeared recently bearing on the application of the Counter Counter to flour analysis (1,2). Particle sizing with this instrument is based on the principle of change in resistivity of a conducting medium in which flour is suspended as the suspension flows through an orifice. The sizing and counting are performed electronically, and the data are subjected to statistical analysis. Analytical precision depends on having a sufficient number of particles sized and counted within each size interval. However, if the particle number at a given interval is excessive, the coincidence effect becomes a problem (3).

In a previous paper from this laboratory (2), a description was given of the particle-size analysis of fractions obtained from the air classification of a 50% extraction soft wheat flour. The mass-median diameters (MMD) of the flours ranged from about 6 to about 40 μ. Distribution analyses of such flours were obtained by a Coulter Counter with orifices of 280 or 140 μ, or a combination of the two.

For a flour with a significantly greater MMD than fine fractions from air-classification separations, such as one obtained by milling wheat with a

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[3]Coulter is a trademark of Coulter Electronics, Inc., Hialeah, Fla. Quadrumat is a trademark of Brabender Instruments, Inc., South Hackensack, N.J. Allis-Chalmers is a trademark of Allis-Chalmers, Milwaukee, Wis. Buhler is a trademark of Buhler Bros., Uzwil, Switzerland. Ro-Tap is a trademark of W. S. Tyler Co., Mentor, Ohio. Alpine is a trademark of Alpine Aktiengesellschaft, Augsburg, West Germany.

Quadrumat Jr. mill (4) or a straight-grade flour from an Allis-Chalmers or Buhler experimental mill, modification in the Coulter Counter method as used in our laboratory become desirable. This paper reports a procedure which utilizes sieving to obtain weight fractions of coarse particles and the Coulter Counter for the distribution of subsieve particles. For the latter, a method is described which permits avoidance of problems associated with insufficient or excessive particle numbers within a given size interval. Data obtained by the two methods (sieving and Coulter Counter) are then integrated to arrive at a particle-size profile of the sample. The procedure described herein may be considered to be an extension of those given previously (2). It permits use of the Coulter Counter in the analysis of relative coarse flours and retains the advantage of measuring fine particles therein.

MATERIALS AND METHODS

Milling

Sets of ten pure-wheat varieties grown at Wooster, Ohio, in the crop years 1963–1966 were used in the experiment. The varieties were Comanche, Purkof, Kawvale, Clarkan, Trumbull, Fairfield, Thorne, Avon (Anderson in 1963), Blackhawk, and American Banner, and represented the range in granularity ordinarily encountered in Eastern soft wheat breeding-line quality-evaluation programs. Wheats were cleaned, conditioned to appropriate moisture contents, scoured, and 2,000-g. batches milled on an Allis-Chalmers laboratory mill, employing a 4-break, 6-reduction system with a finishing sieve of 120-mesh phosphor-bronze screen. Straight-grade and cake patent flours were obtained in separate millings. The latter were milled to 50% (by weight) of the wheat weight, blending streams of the lowest ash contents. The blends were then passed through an Alpine Kolloplex pin-mill at 9,000 r.p.m. and half-feed rate to reduce particle size further.

One hundred fifty-gram subsamples of the same cleaned wheats were conditioned and milled on the Quadrumat Jr. mill provided with a 74-mesh Dur-Loy screen on the reel.

Particle-Size-Index Test

Flour-granulation characteristics of the wheats enumerated above were determined by a particle-size-index test (5), using the Ro-Tap sieving procedure as described in the publication.

Analysis

Sieving. Coarse flours, such as those obtained by milling wheat on a Quadrumat mill, are sieved on a nest of 3-in. sieves with openings of 210, 177, 149, 125, and 105 μ, respectively. The first two sieves are normally omitted when finer flours are analyzed. Sieving is carried out on a Ro-Tap apparatus for 5 min., after which the overs of each sieve are carefully collected and weighed.

An independent experiment was carried out to determine replicability of sieving yield. One hundred fifty-gram wheat subsamples of four varieties (Comanche, Trumbull, Fairfield, and Blackhawk) representing the range in granularity were milled in quadruplicate with the Quadrumat Jr. mill. One-gram subsamples of each flour were sifted in quadruplicate for 5 min. through a set of sieves described above. Earlier observations had indicated that this sieving period was sufficient to ensure good separation. Data were obtained for the quantity of flour held on each sieve but statistical analysis was restricted to the quantity of flour passing through the 105 μ sieve. A nested analysis of variance for flour yield indicated that variety accounted for 96.42% of yield, milling for 1.94%, and sifting for 1.64%. The standard deviation of yield was 0.0054 g., and the least significant difference in yield at the 5% level of probability for a single milling and single sifting of the milled product was found to be 0.025 g. These data showed that the conditions for sifting presently employed are giving replicable data.

The Coulter Counter. Subsieve particle-size-distribution analysis is carried out on the instrument (in our case, Model A), in the normal manner, using a 280 μ orifice and an isopropyl alcohol vehicle containing 4% ammonium thiocyanate (2). A two-stage counting procedure is used: first, a fairly high concentration of flour particles is sized from 95 to 30 μ; and second, a suspension of lower concentration is sized at 40, 35, 30, and lower micron settings at predetermined intervals. The actual quantities of flours used in the determinations are varied according to the granularity of the sample. In the case of the throughs of the 105 μ sieve from Quadrumat-milled soft wheat flours, the flour quantity for the dilute suspension is in the order of 5 mg. per 100 ml. of suspending solution. The concentrated suspensions of the same flour would contain 30 to 40 mg. per 100 ml. The significance of the coincidence factor is the principal criterion in determining the amount to be used for the dilute suspension; very finely divided flour from extremely soft wheat varieties necessarily must be used in even smaller quantities than the 5 mg. mentioned above, whereas the quantity could be increased to 10 mg. for the harder variety flours. Since Allis-milled flours are finer than Quadrumat-milled flours, its sample quantity should also be adjusted to minimize the coincidence-factor effect. Of course, these restrictions do not apply to samples at the higher concentrations, since counts for them are made only to about 30 μ diameter only, at which level coincidence factor and background effects are usually insignificant. It should be noted that these effects often affect counts at low micron settings (for the dilute suspensions), and the counts should be corrected as appropriate.

Computation

The Coulter Counter sizings at 40, 35, and 30 μ at the two concentrations are used to obtain an average concentration ratio for the two suspensions. The counts for the dilute suspension are then multiplied by the concentration ratio and the cumulative-weight values are computed, utilizing a particle-volume factor for each size interval (Table I). Values of these factors are functions of the size interval and the orifice constant.

The $\Sigma(\Delta n)\bar{t}$ value for the largest particles counted by the Coulter apparatus (30341 in Table I) is proportional to the weight of flour analyzed by the Coulter. Weight percentages of smaller particles in each size interval are proportional to $(\Delta n)\bar{t}$ within the interval. Sieved fraction weights are converted directly to cumulative-weight percentage and to $(\Delta n)\bar{t}$ within each size interval.

Values of $t^{2/3}$ for each sieved interval may be computed and included in the calculation for average volume-surface particle diameter $(\overline{d_{vs}})$ (2). The latter forms the basis for flour specific-surface computations, carried out for certain flours in accordance with the procedure described by Wilson et al. (6). All of the above

TABLE I. CUMULATIVE WEIGHT PERCENT OF QUADRUMAT-MILLED THORNE FLOUR BASED ON COMBINED SIEVING AND COULTER COUNTER PARTICLE-SIZE-DISTRIBUTION ANALYSIS

Size Interval μ	Fraction Weight (Sieve) g.	Conc.	Dil.	Adjusted Particle Count	$\bar{t}$[a]	$\Sigma(\Delta n)\bar{t}$[b]	Cumulative Weight[c] %
210–249	0.0243				2,836	60,108	100.0
177–210	0.0921				1,700	58,560	97.4
149–177	0.1364				1,017	52,690	87.7
125–149	0.0961				604.1	43,998	73.2
105–125	0.1182				357.3	37,873	63.0
95–105		4.0		4.0	231.5	30,341	50.5
85–95		11.0		11.0	169.2	29,415	48.9
75–85		37.3		37.3	119.2	28,231	47.0
65–75		55.0		55	80.25	25,096	41.8
55–65		113		113	50.75	23,676	39.4
45–55	0.4761[d]	184		184	29.50	20,732	34.5
40–45		253	39.0	260[e]	18.00	18,637	31.0
35–40		339	55.3	358[e]	12.51	17,269	28.7
30–35		583	74.5	546[e]	8.160	16,043	26.7
25–30			174	1,186	4.933	14,509	24.1
20–25			453	3,087	2.695	11,352	18.9
15–20			1,027	6,998	1.276	6,229	10.4
10–15			1,342	9,144	0.477	1,239	2.1
7.5–10			1,532	10,039	0.166	215	0.4

[a]Calculated relative volume factor for particles within a size interval.

[b]Cumulated product of volume factor and particle number in interval. For sieved fraction, values are proportional to particle weight within each interval.

[c]Assumes uniform particle density for all size intervals.

[d]Weight of flour passing through 105 μ sieve. Only a small portion of this flour was used for the Coulter Counter analysis, as described in the text.

[e]Average ratio of particle count was 6.814 to 1.

computations are made by a computer, which is programmed to require only the sieve fraction weight and raw Coulter-count data as inputs.

Plotting

At the particle-size interface between sieved and Coulter Counter-analyzed fractions (in Table I, between the intervals 105 to 125 μ and 95 to 105 μ), plotting of the cumulative-weight percentages often gave a distorted curve. This was attributed to inefficient sieve separation which resulted in imperfect truncation (7) and, consequently, an underestimated particle count by the Coulter Counter. Past experience indicated that a smooth curve extending from the sieve cumulative-weight percentages could be drawn to a subsieve datum which corrected the distortion.

The mass-median diameter and the 80%-range data may be readily obtained from a smoothed plot of cumulative-weight data. The 80% range is the diameter limits, which include the central 80% of the weight of the particles and is an index of distribution dispersion. The two parameters give a brief and concise description of the particle-size characteristics of a flour.

RESULTS

The proposed sieve plus two-stage Coulter Counter particle-size analysis was applied to Quadrumat- and Allis-milled straight-grade flours, obtaining data

TABLE II. PARTICLE-SIZE AND SIZE-DISTRIBUTION DATA FOR FLOURS BY THREE MILLING TREATMENTS OF TEN VARIETIES GROWN AT WOOSTER, OHIO, 1963–1966 CROPS

Variety	Wheat PSI	Quadrumat		Allis Straight Grade		Pin-Milled Allis Patent	
		MMD	80% range	MMD	80% range	MMD	80% range
	%	μ	μ	μ	μ	μ	μ
1963 Crop							
Comanche	10.4			83	33–117	42.0	18.8–72
Purkof	9.2			80	34–117	38.5	16.3–68
Kawvale	11.4			77	25–113	31.7	16.7–65
Clarkan	11.9			69	22–113	28.7	15.5–60
Trumbull	16.5			56	19–111	26.0	14.0–56
Fairfield	18.2			58	20–108	25.5	14.3–52
Thorne	18.2			62	19–108	24.5	14.7–52
Anderson	18.5			61	20–110	25.0	14.2–52
Blackhawk	14.8			68	20–112	25.0	14.2–50
Amer. Banner	15.4			64	22–108	26.0	15.0–56
1964 Crop							
Comanche	7.9	112	40–183	80	28–112	35.5	17.3–68
Purkof	9.6	112	30–186	76	25–122	27.7	15.3–60
Kawvale	12.4	102	22–183	69	21–115	26.3	14.7–60
Clarkan	13.2	94	22–183	59	19–115	26.5	15.3–58
Trumbull	15.4	102	19–182	51	18–107	23.7	13.6–51
Fairfield	21.2	84	19–185	34	17–103	24.5	13.8–49
Thorne	18.2	94	19–182	46	17–108	23.8	14.0–46
Avon	15.5	90	20–180	63	18–117	24.0	13.5–52
Blackhawk	14.4	102	21–180	62	20–112	24.5	14.5–50
Amer. Banner	16.9	92	20–183	60	20–113	24.5	14.3–49
1965 Crop							
Comanche	9.6	112	33–190	88	33–112	39.0	16.7–70
Purkof	10.5	108	33–183	85	29–121	36.5	15.3–73
Kawvale	12.7	100	23–185	74	23–118	29.0	15.4–63
Clarkan	12.0	103	24–185	73	22–119	30.0	14.8–63
Trumbull	14.7	94	19–192	63	19–117	24.8	14.0–55
Fairfield	16.5	85	20–178	67	19–120	24.5	13.5–55
Thorne	15.3	92	20–183	64	20–117	24.7	14.5–54
Avon	14.6	96	22–183	72	22–117	28.5	15.0–60
Blackhawk	15.3	100	21–178	73	21–117	25.7	13.6–57
Amer. Banner	14.6	100	21–180	72	22–117	26.5	14.3–56
1966 Crop							
Comanche	10.8	106	26–195			28.0	13.8–66
Purkof	9.7	112	28–200			31.0	14.5–65
Kawvale	14.4	95	21–197			24.5	12.8–59
Clarkan	14.3	100	22–190			24.5	13.0–54
Trumbull	22.0	81	18–188			21.0	11.1–42
Fairfield	22.2	71	18–180			22.8	12.1–46
Thorne	21.4	80	18–188			21.8	12.0–45
Avon	18.4	86	19–188			22.5	12.5–54
Blackhawk	16.3	92	19–183			23.0	13.0–53
Amer. Banner	19.1	80	19–185			23.5	13.0–53

presented in Table II. Analysis of the pin-milled Allis patent flours, made by using in combinations of 280 and 140 μ orifices on the Coulter Counter (but bypassing the sieving) are also given in the same table. Ten-variety mean MMD values for Quadrumat-, Allis-straight grade, and pin-milled Allis patent flours were 96.0, 67.0, and 27.1 μ, respectively. Considering only the seven soft wheats (omitting data for

Comanche, Purkof, and Kawvale), corresponding MMD values were 91.3, 61.8, and 24.9 μ, respectively. These latter values may be regarded to be representative of soft-wheat flours milled by the above methods.

Average 80% ranges were 22.5 to 185.3 μ, 22.2 to 113.9 μ, and 14.4 to 56.7 μ, respectively, for flours of all ten varieties obtained by the three milling procedures, respectively. For the seven soft wheat varieties, the values were 20.0 to 183.7 μ, 19.8 to 112.8 μ, and 13.8 to 52.9 μ, respectively. These data show that a significant portion of Allis-milled straight-grade soft wheat flour is larger than 105 μ, the size readily counted with precision by using the 280 μ orifice, and pointing to the advantage of combining the Coulter with a sieving procedure.

Correlation coefficient data (Table III) indicate that among the milling procedures Quadrumat, Allis-straight grade, and Allis patent pin-milling, wheats with certain inherent granulation properties maintain their relative fracturing characteristics for a given type of milling. This relative endosperm friability of varieties is also revealed by a simple wheat-grinding technique such as the particle-size-index test. It is interesting to note that varietal resistance or susceptibility of endosperm to fracturing extends to pin-milling.

TABLE III. CORRELATION COEFFICIENTS FOR RELATIONSHIPS AMONG PARTICLE SIZE INDEX, MMD FOR QUADRUMAT, ALLIS-CHALMERS STRAIGHT GRADE, AND ALLIS-CHALMERS PATENT (PIN-MILLED) FLOURS, FOR TEN VARIETIES[a]

	MMD Quadrumat Flour	MMD Allis Straight Grade	MMD Allis Patent
Particle Size Index	−0.936[b] −0.929	−0.851[b] −0.914	−0.783[c] −0.777
MMD Quadrumat		0.714[d] 0.815	0.750[b] 0.736
MMD Allis Straight Grade			0.766[b] 0.768

[a]Grown at Wooster, Ohio, 1963–1966 seasons. Within each pair, upper value is for pooled data, lower is for same data adjusted for crop year.

[b]Data for 3 crop years (n = 30).

[c]Data for 4 crop years (n = 40).

[d]Data for 2 crop years (n = 20).

All coefficients are significant at the 0.1% level of probability.

DISCUSSION

The procedure described above for the combined sieve and Coulter Counter particle-size-distribution analysis of coarse-ground flour, and the usual Coulter procedures for more finely ground materials, make possible the characterization of flour milled in a variety of ways. The combined sieve and Coulter Counter method for particle-size-distribution analysis is effective in obtaining profiles on soft wheat flours with MMD in excess of about 50 μ. Flours with this or greater MMD normally will have the upper limit of the 80% range in the particle-size range at or greater than 110 μ. The 280 μ orifice of the Coulter Counter does not readily accommodate particles much larger than 95 μ, hence sieving is appropriate.

For flours with MMD in the range of 15 to 50 μ, the Coulter Counter only, with an orifice of 280 μ diameter or combination of 280 μ and 140 μ, may be used with advantage, whereas finer flours with MMD less than 15 μ (such as certain fine cuts obtained in air classification) may be analyzed using the 140 μ orifice only. These procedures were described briefly elsewhere (2).

The computer used in calculating the cumulative-weight data for a flour is also programmed to calculate the volume-surface mean diameter of the distribution and the specific surface value for the flour. The latter is related to MMD, but is influenced by type of milling. Thus, at the same MMD (Fig. 1), Quadrumat-milled flours had greater specific surfaces than Allis-milled straight-grade flours. The coarse particles in the Quadrumat flours apparently influenced MMD more than they did specific surface.

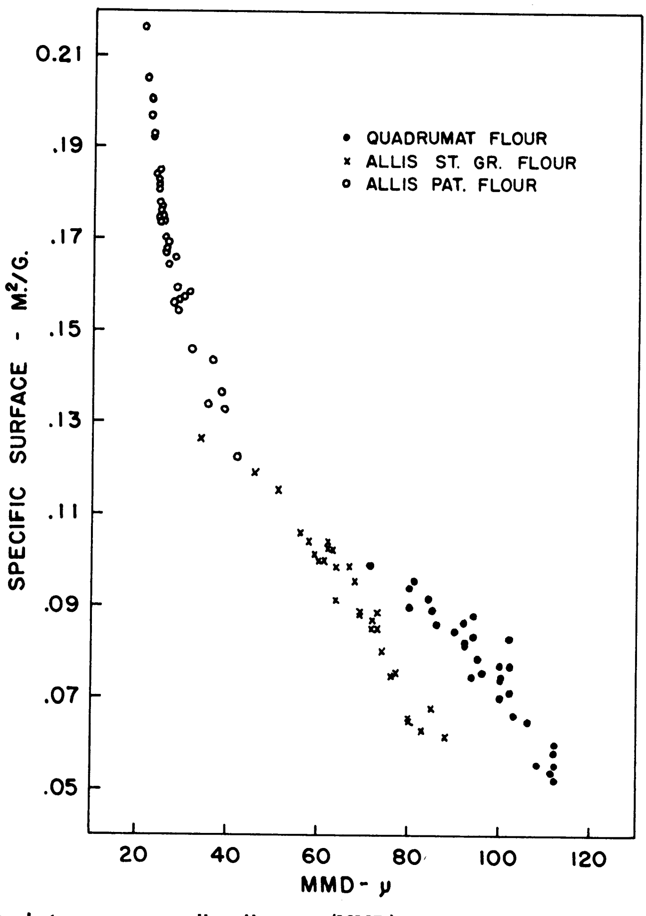

Fig. 1. Relation between mass-median diameter (MMD) and specific surface for Quadrumat (3 crop years), Allis-Chalmers straight-grade (3 crop years), and pin-milled Allis-Chalmers patent flours (4 crop years).

Particle-size distribution of fractured endosperm in wheat appeared to be predominantly a varietal characteristic, affected to only a limited extent by season of growth. As the degree of grinding was increased, the association between variety and MMD tended to become less precise, indicating the progressive entrance of grinding variables.

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