MEASUREMENTS OF THE PARTICLE SIZE DISTRIBUTION OF FLOUR[1]

R. R. IRANI[2] AND W. S. FONG[3]

ABSTRACT

The particle size distribution of flour, measured microscopically by gravitational sedimentation, centrifugal sedimentation, and sieving, all gave mean diameters and distribution curves which agreed with one another within experimental error. Measurements using changes in electrolytic resistivity gave particle size distributions that deviated significantly and nonrandomly from the other methods.

The effect of particle size distribution on the quality of flour, including the ratio of starch to protein content, has been known for many years, and studies are still being carried on (8). Recently, interest in particle size distribution measurements has mushroomed, and several new techniques and commercial instruments have been developed.

Sieving, sedimentation, and photoextinction techniques have received most of the attention in the measuring of particle size distribution of flour. Whitby (24,26) and Heywood (12) discussed the mechanics of fine sieving and concluded that sieving can be divided into two different steps. During the first step, particles with a size

[1] Manuscript received January 19, 1960.
[2] Research Department, Inorganic Chemicals Division, Monsanto Chemical Company, St. Louis 66, Mo.
[3] Central Research Laboratory, DCA Food Industries, Inc., New York 18, N. Y.

much smaller than the sieve opening pass through. During the second and relatively slower step, particles whose size is close to that of the opening are sieved through.

Kent-Jones *et al.* (18) describe a simple sedimentation procedure for determining flour granularity. Heald (9) used a mixture of carbon tetrachloride and naphtha as the sedimentation fluid and utilized an adaptation of the Andreasen method (3) to measure the particle size distribution of flour.

The photoextinction technique has received much attention. Kent-Jones *et al.* (18,19) described a photosedimentation method in which the percentage of different-sized particles in flour is correlated with the decrease in cloudiness with time. Amos (2), using this photosedimentation method, concluded that small changes in the granularity of flour do not affect its quality for biscuit production. All known photoextinction methods, whether sedimentation is involved or not, are questionable because of the large errors (21) involved due to dependence of the extinction coefficient on particle size and shape factors (20). For example, Deschreider (7) found that the turbidity of flour suspensions rose with decreasing particle size and increasing ash content even when the flour concentration in the suspension was kept constant.

Hildebrand *et al.* (11) discussed the advantages and disadvantages of microscopic determinations of particle size of flour; they also concluded that sieving and sedimentation become inaccurate below 200 mesh (74μ) and above 60μ respectively. On the other hand, Wichser and Shellenberger (27) compared Ro-Tap sieving with elutriation and sedimentation (Andreasen) and concluded that sieving was most applicable above 37μ. Whitby (23) discussed various methods of measuring the particle size distribution of flour and pointed out that some of the inconsistencies are probably due to differences in particle shape and density.

In discussing particle size distributions, it is essential to define "size" for a particle with an irregular shape. If the center of gravity of an irregular particle is located, an extremely large number of lines terminating at the edges of the particle but passing through the center can be drawn. These lines are diameters of the particle. The average of these diameters, either arithmetic or geometric, will be used as the definition of particle size[4] throughout this paper.

This paper will present a comparison of various methods of measuring particle size distribution of soft wheat flour. The methods

[4] Irani, R. R., Ames, D. P., and Callis, C. F. Unpublished results, Monsanto Chemical Company, Inorganic Chemicals Division, St. Louis 66, Mo.

comprise microscopy, gravitational sedimentation, centrifugal sedimentation, sieving, and changes in the electrolytic resistivity.

Methods that give only an average size and no distribution will not be considered, because in most cases these so-called averages do not adequately represent the entire size distribution and are often affected by properties of the powder other than size, e.g., permeability measurements (13).

Materials and Methods

Materials. Ten commercially milled soft wheat flours were employed. A commercial grade of tricalcium phosphate, having a geometric mean weight size of 2.3μ (98% between 0.3 and 7μ) with a calcium oxide to phosphorus pentoxide molar ratio of 3.18 and exhibiting an apatite X-ray pattern (22), was added to the flour when dry sieving was utilized.

Gravitational Sedimentation. The sedimentation measurements were made with a Monsanto-automatized Bostock type (5) liquid sedimentation balance, previously described (1). The dispersions were obtained by the gradual dilution of the weighed sample of flour, and examined microscopically to verify the deagglomeration of the particles. The volume fraction of the powder in the dispersion was always kept below 0.3% to ensure free settling.

The liquid sedimentation data for an initially homogeneous suspension were interpreted to give particle size distributions by the methods outlined by Oden and modified by Bostock (5):

$$w = W - \frac{dW}{d\ln t} \qquad (1)$$

where W is the settled weight at time, t, and w is the weight of that fraction of settled particles whose size, M, would allow sedimentation through the entire height of the column, h, as given by Stokes' equation

$$M = \sqrt{\frac{18\,\eta\,h}{(\rho_1 - \rho_2)\,g\,t}} \qquad (2)$$

where η and ρ_2 are the viscosity and density of the sedimentation fluid, respectively, and g the acceleration due to gravity .The particle density, ρ_1, was measured pycnometrically (6).

Flour is known to be a heterogeneous mixture of particles of different densities and shapes. Duplicate specific gravity determinations of five flour samples and their air-classified fractions gave values ranging from 1.40 to 1.50 with an average value of 1.44. The

TABLE I

PARTICLE SIZE DISTRIBUTION BY WEIGHT FOR A SAMPLE OF FLOUR FROM SEDIMENTATION IN VARIOUS SOLVENTS

Size	Percent by Weight Greater than			
	Ethanol	Benzene	Isobutanol	2-Ethyl Hexanol
μ				
5	100	99.5	99.5	99.2
10	98.1	96.2	97.8	95.1
20	80.0	83.0	85.1	84.9
30	65.1	70.9	71.2	68.7
40	44.0	49.0	46.0	48.0
50	37.0	36.0	38.0	39.0
60	30.0	29.5	28.0	30.0
80	17.0	19.0	20.0	18.0
100	5.0	4.0	7.0	7.0
120	0.0	1.0	0.0	1.0

previously defined are some averages of the diameters of the particles. The arithmetic and geometric averages, known to give the largest difference between averages, have recently been shown (see footnote 4) to differ by only 6.0% for a particle whose largest diameter is three times the smallest diameter. This finding justifies the intercomparison of various methods for particle size distribution measurements of flour.

Even though ten flour samples were investigated, for the sake of brevity the results are presented on only two samples. The particle size distributions obtained by the various methods for two flours with geometric mean particle sizes of about 45 and 8μ are given in Tables II and III respectively.

The centrifugal sedimentation method was not used on the coarser sample because the lowest r.p.m. centrifuge caused substantial amounts of sediment to settle in a matter of seconds.

Only incomplete sieving data are presented on the finer flour sample because no sieves have been manufactured in the size range below 10μ.

For both samples of flour, all investigated methods agree with one another within experimental error except for the Coulter counter, which gave results systematically different from microscopy and sedimentation. This discrepancy has been discussed by Irani and shown to be resolvable (15) for flour provided the Coulter counter is calibrated with flour rather than with an arbitrary powder. In agreement with previous work (1) the data obtained by microscopy and sedimentation agree with one another in all cases.

In agreement with previous findings (16), the results indicate that the recently developed micromesh sieves, when calibrated, and in combination with a conditioning agent, namely, tricalcium phos-

TABLE II

INTERCOMPARISON OF METHODS ON FLOUR

Size	Percent by Weight Greater than by				
	Microscopy	Sieving [a]	Sieving [b]	Gravitational Sedimentation [c]	Coulter Counter
μ					
5	99.6	..	..	100.0	99
10	98.5	..	..	98.2	95
15	94.0	..	..	93.2	91
20	87.0	..	..	82.0	82
25	81.0	..	..	76.0	74
30	74.0	73	78	68.0	68
35	...	..	..	60.0	62
40	65.0	..	..	54.0	56
45	...	58	63	48.0	52
50	43.0	..	..	38.0	..
55	...	..	..	...	..
60	32.0	42	28	30.0	30
70	...	..	..	20.0	11
80	18.0	18	2	...	1
90	...	..	..	...	..
100	7.0	..	..	5	0
120	0	..	..	0	0

[a] Brushing.
[b] Ro-Tap with 1% tricalcium phosphate.
[c] Average of data obtained in ethanol, isobutanol, and 2-ethyl hexanol.

phate, are accurate down to 15μ, whereas conventional woven sieves become inaccurate below 50μ (1,11). Attempts to calibrate woven sieves below 50μ were unsuccessful.

Figure 1, the plot of the data on log-probability graph paper such as K & E 359-24, reveals that the finer flour sample investigated follows a log-normal distribution, whereas the coarser flour sample investigated represents an abnormal log-normal distribution that had been artificially scalped at 120μ (14).

TABLE III

INTERCOMPARISON OF METHODS ON FLOUR

Size	Percent by Weight Greater than by				
	Microscopy	Gravitational Sedimentation [a]	Centrifugal Sedimentation [b]	Sieving [c]	Coulter Counter
μ					
2	99.6	99.3	..	...	100.0
3	97.5	97.0	95	...	100.0
4	89.0	89.0	..	...	100.0
6	68.0	...	60	...	98.8
8	44.0	46.0	34	...	...
10	29.0	...	20	...	72.0
15	10.0	10.0	6	16.0	45.0
20	3.5	2.0	..	6.0	12.0
30	0.5	...	..	1.2	1.9

[a] Average of data obtained in ethanol, isobutanol, and 2-ethyl hexanol.
[b] Average of data obtained in ethanol and benzene.
[c] Brushing.

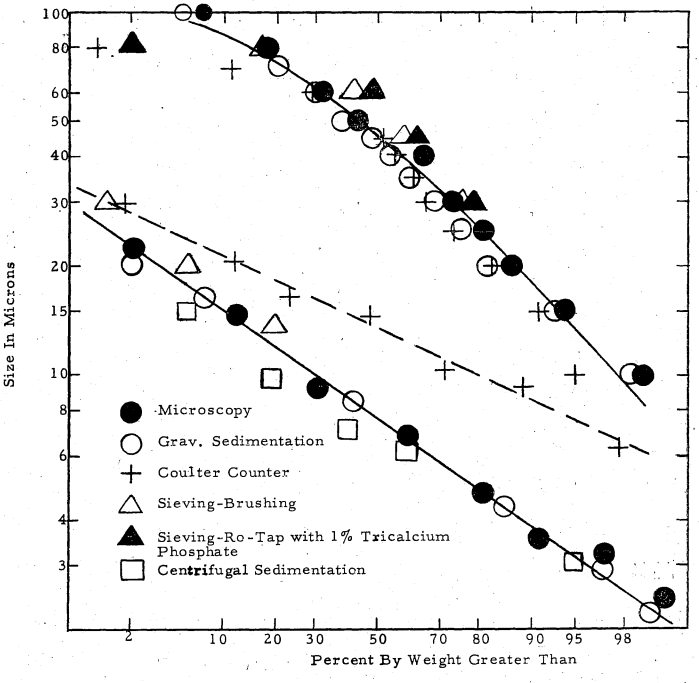

Fig. 1. Intercomparison of methods for measuring the particle size distribution of flour.

Many control tests on flour require a knowledge of the percent finer than or coarser than a *specific size*. When this percentage is less than 10% or larger than 90%, it is obvious, from Fig. 1, that the control limits must be set for each technique independently. While the various methods with calibration or otherwise agree with one another as far as the average size and the shape of the distribution curve are concerned, they give results that are significantly different from one another if only one point on the distribution curve is considered. Therefore, one-point limits, which are very inadequate in describing size distributions, should be set for each method independently. In addition, for these limits to be at all meaningful, they should not approach the precision of the specific technique.

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