

DISTRIBUTION OF ASH AMONG FLOUR EXTRACTS AND FRACTIONS AND ITS RELATION TO ELECTRICAL CONDUCTIVITY^{1,2}

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ABSTRACT

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Flours from soft red, soft white and hard red winter wheats, and defatted and bleached flours were fractionated and ash was determined in the fractions. In addition, various flours were serially extracted five times with water and ash was determined in extracts and residues. Ash was also determined in extracts of millstream flours representing a wide range of ash levels. Electrical conductivities of all extracts were measured. In the fractionations, ash from two aqueous extractions accounted for 50% of total recovered ash, with starch contributing

27-30%, gluten 11-21% and tailings 4-8%. Variations among flours apparently resulted partially from differences in water retention. From fivefold extraction of flours, 72-77% of total recovered ash was in the combined extracts and 23-28% in the residues. From millstream flours suspended in water for one hr at room temperature, recovery of total ash from the liquid phase was 75-80% for low-ash, and 60-65% for high-ash flours. For extracts prepared under uniform conditions, conductivities were proportional to ash levels of flours.

Soluble minerals that contribute to flour ash influence flour behavior in aqueous systems through their effects on protein dispersibility, and influence gliadin levels in flour extracts (1-3). Recently, the author showed that gluten becomes hydrophilic after exposure to high concentrations of salts and used electrical conductivity to monitor salt levels in gluten washings (4). During these studies, an interest was developed in the endogenous electrolytes and their effects on gluten behavior.

Data from several studies relating to specific elements indicate the degree to which inorganic constituents of flour are extracted by water. El Gindy *et al.* (5) studied the elemental compositions of flour fractions in relation to agronomic variables and found 44-51% of the recovered ash in the water extract, 40% in the starch, and 9-16% in the gluten. In a study of distribution of individual elements, Bequette *et al.* (6) reported an average of 40% of the ash in the water extract, 40% in the starch, and 20% in the gluten. Sollars (7) found 60% of the chloride of unbleached flour in the water extract, and 86.5% of the phosphorus of bran was extracted with water (8). The water-soluble solids obtained by aqueous fractionation of flour apparently contain much of the inorganic material. However, the published data probably do not provide a true representation of extractable minerals because a substantial proportion of the soluble material can

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remain in the liquid phase associated with the water-insoluble fractions.

The purpose of this study was to determine mineral (ash) levels in flour extracts prepared under various conditions, and to relate these levels to electrical conductivity. The approaches were: various types of flours were fractionated by a conventional aqueous procedure and ash was determined in extracts and water-insoluble fractions; flours were serially extracted five times, and ash was determined in the extracts and residues; and millstream flours representing a wide range of ash levels were extracted with water and aliquots were ashed. Electrical conductivities of all extracts were measured.

MATERIALS AND METHODS

Flours

Wheat flours in this study were: soft red winter (SR); soft white winter (SW); hard red winter (HR); soft red winter and soft white winter (S); soft red and soft white, hexane extracted (S de-fat); hard red winter (Kans.) (HRW); hard red winter (Kans.), hexane extracted (HRW De-fat); soft 50% patent, pin-milled (S Pat); soft 50% patent, pin-milled and chlorine bleached (S Pat Bl). The Kansas hard red winter wheat flour (HRW) was provided by the USDA Hard Winter Wheat Quality Laboratory at Manhattan, Kans. The soft patent flour was obtained from commercial sources and a sample was treated with chlorine in the laboratory. The remaining flours were straight grade flours from routine millings in the Soft Wheat Quality Laboratory at Wooster. Defatted flours were extracted with hexane for 24 hr in a Soxhlet apparatus.

Fractionation

Duplicate 250-g flour samples were blended with 500 ml water (double deionized, conductivity 0.5–1.0 μ mho/cm) for 2 min and centrifuged at 1,000 \times *g* for 20 min. The supernatant (first extract) was collected and the volume measured, and an equal volume of water was added back to the residue. The mixture was again blended and centrifuged and the supernatant (second extract) was collected. Tailings, gluten and starch were separated and freeze-dried. Conductivities of the supernatants were determined, and the extracts were freeze-dried. Dried products were weighed and ash was determined gravimetrically after incineration at 555°C for 15 hr.

Fivefold Extraction

The extraction procedure was the same as for fractionations, except that blending and centrifugation were repeated to give a total of five serial extractions. The residues were not fractionated, but were homogenized in water and freeze-dried. Conductivities of the extracts were determined and the extracts were freeze-dried. Dried products were weighed and analyzed for ash.

Millstream Flour Extractions

Millstream flours were from a milling of a soft red winter wheat (Logan) on a MIAG Multomat mill, and consisted of streams from three breaks, five reductions, a duster, and a grader. Five g flour was suspended in 20 ml double-deionized water in 50-ml capped tubes for 1 hr and shaken frequently. The

suspensions were centrifuged at $10,000 \times g$, supernatants were decanted and conductivities measured, and 5-ml aliquots were dried and ashed. Residue ash was determined by subtracting soluble ash from flour ash.

Conductivity Measurements

Samples of extracts were boiled and cooled. Conductivities were measured at 25°C in a Radiometer Type CDC114 pipet-type cell (1 ml) with a Radiometer Type CDM2e conductivity meter. Measurements were corrected for cell constant and all data refer to specific conductivity and/or specific resistance.

RESULTS AND DISCUSSION

Data from the fractionation experiments are summarized in Tables I and II, and in Fig. 1. In the eight flours fractionated, approximately 50% of the total recovered ash was in the two water extracts, 27–30% was in the starch, 11–21% in the gluten, and 4–8% in the tailings (Fig. 1). These values agree in general with published reports (5,6). Presumably, more than 50% of the ash was solubilized, but some mineral matter was retained in the liquid remaining in the solid fractions. The slight differences in ash distribution among the eight flours probably reflect differences in water retention, as well as in extractability or mineral content. A strongly hydrophilic flour (such as HRW De-fat) under these conditions might appear to have low extractable ash due to its low volume of supernatant.

The extracted solids generally yielded about 5% ash (dry basis), but solids from extracts of bleached flour yielded only 1–2% ash, presumably because of high gluten extraction (due to low pH) and consequent dilution of solids (Table II). However, total ash was similar in extracts of bleached and unbleached flours (Table I).

TABLE I
Ash Distribution Among Extracts and Solid Fractions after Wet
Fractionation of Eight Flours (Averages of Duplicates)^a

Flour	Ash ^b , %					% of Total Recovered Ash					Total Ash % of Flour	
	Ext. 1	Ext. 2	Tail.	Glut.	Starch	Ext. 1	Ext. 2	Tail.	Glut.	Starch	Sum ^c	flour ^b
S	5.05	4.91	0.65	0.29	0.23	31.3	19.3	6.3	12.5	30.6	0.42	0.44
S De-fat	4.46	6.10	0.69	0.40	0.21	25.5	20.8	7.2	19.4	27.1	0.43	0.44
HRW	5.17	5.10	0.89	0.47	0.23	25.7	21.4	6.2	18.2	28.5	0.47	0.49
HRW De-fat	4.69	4.41	0.92	0.44	0.25	22.8	18.7	8.3	20.7	29.5	0.48	0.49
SR	5.84	3.47	0.65	0.42	0.20	36.6	9.8	5.9	17.3	30.5	0.41	0.44
SW	5.59	2.73	0.52	0.37	0.20	38.0	10.1	5.8	16.7	29.4	0.40	0.42
S Pat	4.13	2.87	0.48	0.28	0.17	32.6	20.8	4.5	11.3	30.9	0.34	0.32
S Pat B1	2.05	1.20	...	0.44 ^d	0.13	32.6	26.1	...	14.0 ^d	27.4	0.31	0.33

^aFlour:water = 1:2.

^bDry weight basis.

^cObtained by summation of values from individual fractions.

^dGluten and tailings combined.

TABLE II
Ash Content and Specific Conductivities of First and Second Extracts of
Eight Flours (Averages of Duplicates)^a

Flour	First Extract				Second Extract			
	Solids per 100 ml g	Ash in solids ^b %	Ash per 100 ml g	Conduct'y m mho/cm	Solids per 100 ml g	Ash in solids ^b %	Ash per 100 ml g	Conduct'y m mho/cm
S	1.97	5.05	0.10	1.47	1.07	4.91	0.05	0.79
S De-fat	2.06	4.46	0.09	1.50	1.09	6.10	0.07	0.97
HRW	2.39	5.17	0.12	1.84	1.65	5.10	0.08	1.26
HRW De-fat	2.52	4.69	0.12	1.79	1.82	4.41	0.08	1.26
SR	2.03	5.84	0.12	1.79	0.98	3.47	0.03	1.09
SW	2.01	5.59	0.11	1.66	1.10	2.73	0.03	0.93
S Pat	1.93	4.13	0.08	1.31	1.67	2.87	0.05	0.67
S Pat B1	4.75	2.05	0.10	1.67	4.65	1.20	0.06	0.98

^aFlour:water = 2:1.

^bDry weight basis.

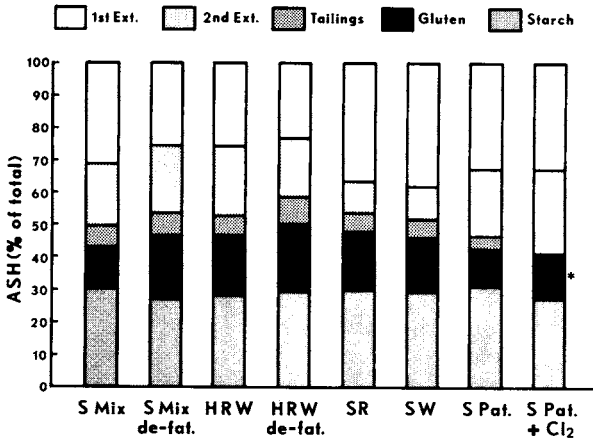


Fig. 1. Distribution of ash among extracts and solid fractions from wet fractionation of eight flours. (See text for key to flours). Flour:water = 1:2.

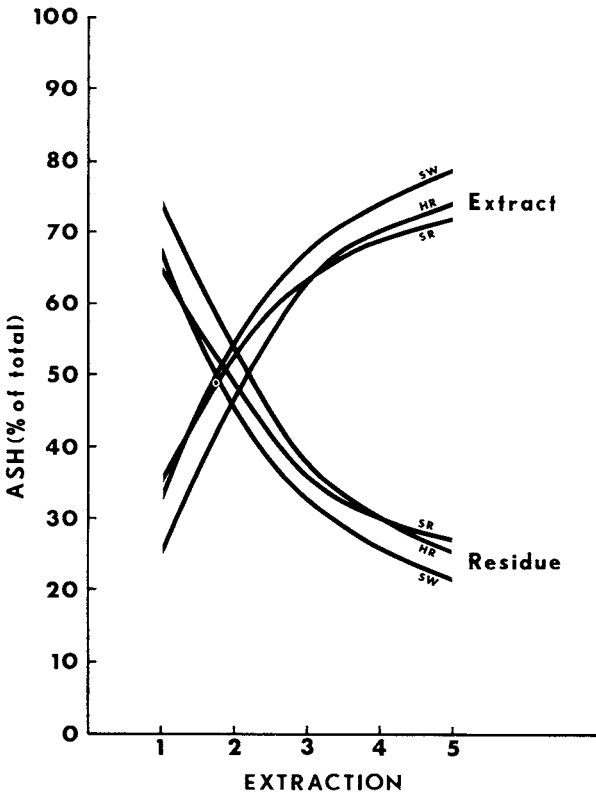


Fig. 2. Ash in extracts and residues during fivefold extraction of three types of flours (SR = soft red; SW = soft white; HR = hard red). Flour:water = 1:2.

Results of fivefold extractions of SR, SW, and HR flours are shown in Table III and Fig. 2. Total ash from five extractions accounted for 72–77% of total recovered ash (as compared with 50% from two extractions), with 25–35% in the first extract and 4% in the fifth extract. Extraction was slowest for flour HR, possibly because of its vitreous character, but also because of its higher water retention (Fig. 2). During the sequence of extractions, ash levels in the extracted solids from each flour increased to a maximum (about 6% for SW and HR, and 7% for SR) in the second or third extract, and declined in the subsequent extracts (to 2–4% in the fifth extracts). The declines in ash and in solids during the extraction sequence suggest that further extraction would recover only negligible ash. The residue after five extractions constituted more than 90% of total recovered solids,

TABLE III
Ash Content and Specific Conductivities of Five Extracts of Three Types of Flours (Averages of Duplicates)^a

Extract	Volume ml			Solids ^b g/100 ml			Ash ^b g/100 ml			Conductivity m mho/cm		
	SR	SW	HR	SR	SW	HR	SR	SW	HR	SR	SW	HR
1	313	330	243	2.01	1.85	2.19	0.13	0.10	0.10	1.78	1.63	1.67
2	319	320	275	0.99	1.15	1.36	0.06	0.07	0.08	0.88	0.90	1.21
3	325	320	290	0.49	1.17	0.76	0.04	0.04	0.05	0.52	0.49	0.72
4	325	325	300	0.44	0.71	0.40	0.02	0.02	0.02	0.30	0.25	0.39
5	325	325	300	0.30	0.49	0.25	0.01	0.01	0.01	0.16	0.14	0.23

^aFlour:water = 1:2.

^bDry weight basis.

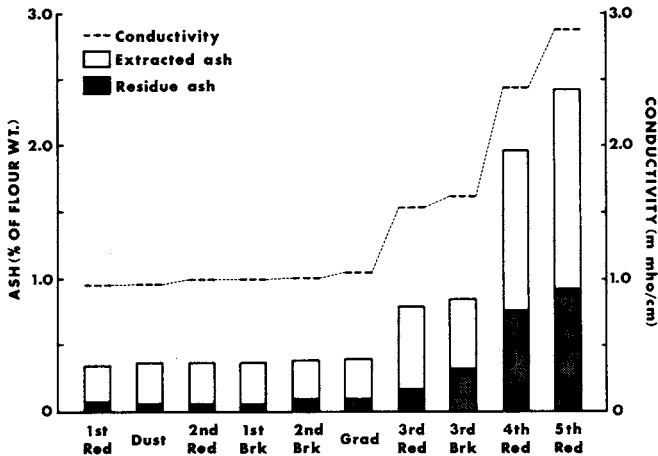


Fig. 3. Aqueous extraction of ten millstreams from a milling of a soft red wheat (Logan), showing ash in extracts and residues in relation to specific conductivities of the extracts. Flour:water = 1:4. (Red = reduction; Dust = duster; Brk = break; Grad = grader).

but less than 25% of total ash. The extracted solids, on the other hand, constituted less than 10% of the total solids, but contributed 75% of the ash.

For the series of millstream flours from a soft red wheat (Logan), calculation of total extracted ash was based on total liquid, rather than on solids from the decanted supernatant (Fig. 3). Therefore, the levels, which represent total "solubilized ash" and include ash in the liquid retained by the residue, are true indications of ash in the total liquid phase after equilibration for one hr at room temperature. Under those conditions, 60–80% of the total flour ash was found in the liquid phase. About 75–80% of total flour ash was accounted for by ash from extracts of low ash streams (less than 1% ash), while 60–65% of the total ash from high-ash streams (more than 1% ash) was recovered from the liquid phase.

Electrical conductivities were measured in each of the foregoing procedures and are included in the data (as specific conductivity). Table II shows values for the two extracts from each fractionation (eight flours) and Table III shows conductivities of extracts from the more exhaustive fivefold washings (three flours). In general, conductivities paralleled ash values; the range decreased from 1.6 to 1.8 m mho/cm for the first extracts (equivalent to 0.01 M NaCl) to 0.1 to 0.2 m mho/cm (equivalent to 0.001 M NaCl) for the fifth extracts. The conductivities of the millstream flour extracts in Fig. 3 also paralleled ash levels. Additional data that show the relation between conductivity and ash are presented elsewhere.

The data indicate that exhaustive aqueous extraction of flour solubilizes material that contributes more than 80% of the total flour ash, but conventional aqueous fractionations give a water-soluble fraction that contributes only about 50% of the total flour ash. Recovery of ash from high-ash flours (ash > 1%) was lower than from low-ash flours. The preliminary results suggest that conductivity measurements of flour extracts might be useful for estimation of the levels of electrolytes and of total ash in flours.

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