

# CHEMICAL, PHYSICAL, AND NUTRITIONAL PROPERTIES OF HIGH-PROTEIN FLOURS AND RESIDUAL KERNEL FROM THE OVERMILLING OF UNCOATED MILLED RICE. III. IRON, CALCIUM, MAGNESIUM, PHOSPHORUS, SODIUM, POTASSIUM, AND PHYTIC ACID<sup>1</sup>

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## ABSTRACT

Six lots of uncoated, commercially milled rice of different varieties were abraded in a rice-polishing machine, and the samples were analyzed for iron, calcium, magnesium, phosphorus, sodium, potassium, and phytic acid. An additional six lots were studied for iron only. First-pass flours (about 2% of the kernel) contained 23 times as much phytic acid as did the original rice; iron, 14 times; magnesium, 11; phosphorus, 9; calcium and potassium, each 6; and sodium, 1.5. Concentrations of these components decreased with each successive pass, while the residual kernel contained less than did the original rice. Residual kernels, about 87% of the original rice, contained 72% as much

sodium as was found in the whole kernel; 63 to 69% as much potassium, phosphorus, and calcium; 34% as much iron; 30% as much magnesium; and no detectable phytic acid. Phytic acid phosphorus accounted for about 30% of the total phosphorus in the original rice, but for 70 to 80% of the total phosphorus in the flours. Sodium was the mineral most evenly distributed throughout the kernel; phytic acid showed the steepest gradient. The flours, about 6% of the original kernel, accounted for nearly all of the phytic acid, 73% of the iron, 50% of the magnesium, 38% each of ash and phosphorus, 25% each of calcium and potassium, but only 8% of the sodium.

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Although much information is available concerning the content of nutrients in the various anatomical parts of seed kernels, relatively little can be found on their distribution within the endosperm. For rice, only a few data on minerals in different parts of the endosperm have been published. Houston et al. (1) reported concentrations of ash in rice flours abraded in a CeCoCo mill, from a lot of commercially milled Calrose rice, that were seven times as high as in the whole rice, with percentage of ash decreasing as the center of the kernel was approached. Similar data were reported by Hogan et al. (2) for three lots of long-grain rice in which the values for ash in flours from the outer layers were 10 to 20 times as high as for those in the whole kernels. Normand et al. (3) found high concentrations of phosphorus and calcium in the outer fractions obtained by the abrasive milling of a lot of conventionally milled, long-grain rice. The concentrations were also 10 to 20 times that of the whole rice, and decreased from the periphery to the center.

As part of a study on the properties of rice flours obtained by the abrasive milling of commercial rice, the concentrations of phytic acid and of the mineral constituents iron, calcium, magnesium, phosphorus, sodium, and potassium were determined. Results of the analyses on whole kernel, three abraded flours, and residual kernel of six varieties of rice are reported here.

#### MATERIALS AND METHODS

The rice samples and milling procedure have been described previously (4). Minerals and phytic acid were determined on the six varieties of 1966 rices: for each lot, original and residual kernels and three flours which passed through a 40-mesh screen. All 12 lots of rice, however, were analyzed for iron. All determinations were made in triplicate and are reported on a moisture-free basis.

Iron was determined with 2,2'-bipyridine, which forms a deep red complex with ferrous salts in acid solution (5). Whole and residual kernels were finely ground in a mill with stainless steel cutters, to minimize contamination with iron, and percent moisture was determined on these specially ground samples. Samples were ashed in Vycor crucibles.

For calcium and magnesium, ashes were treated with concentrated hydrochloric acid, the acid was evaporated, and the residues were baked at 100°C. for 4 hr. to render the silica insoluble. After the residue was dissolved in acid, the filtrate was analyzed for calcium by precipitation with ammonium oxalate and titration with potassium permanganate (6). Magnesium was determined by EDTA titration (7).

Phosphorus was determined by reading the blue color of the phosphomolybdate complex in a photoelectric colorimeter (8). Sodium and potassium were analyzed by atomic absorption spectroscopy (9). Phytates, reported as phytic acid, were determined by extraction of the sample with trichloroacetic acid solution, precipitation of the phytic acid as ferric phytate, decomposition of the ferric phytate precipitation with sodium hydroxide, and colorimetric determination of the iron by means of sodium salicylate, according to the method of de Lange et al. (10).

#### RESULTS

As a check on our analyses, the sum of the components in the milled fractions

TABLE I  
Iron Content of Fractions of Overmilled Rice  
(mg. per 100 g., dry basis)

Rice Lot and Variety <sup>a</sup>	Whole Kernel	Flour through 40-mesh Screen			Residual Kernel after Third Pass
		First Pass	Second Pass	Third Pass	
1966					
BP	1.48	22.7	17.1	14.4	0.36
BB	0.45	8.5	5.8	5.4	0.15
CR	0.71	14.0	5.7	3.3	0.19
S	0.42	6.2	2.8	2.4	0.13
CL	0.26	12.1	7.3	5.1	0.14
CS	0.37	5.6	2.4	2.1	0.17
Avg.	0.62	11.5	6.8	5.4	0.19
Std. dev.	±0.45	± 6.4	± 5.4	± 4.6	±0.09
1968					
CB	2.34	35.6	12.1	6.7	0.30
CC	0.88	11.0	4.7	2.1	0.31
TE	1.75	23.4	9.1	5.2	0.26
TL	1.62	17.9	8.3	7.8	0.38
CP	1.08	9.3	6.2	3.4	0.48
TP	1.17	14.6	12.5	8.0	0.93
Avg. (n = 12)	1.04	15.1	7.8	5.5	0.32
Std. dev.	±0.65	± 8.7	± 4.3	± 3.5	±0.22

<sup>a</sup>BP, Texas Belle Patna; BB, Arkansas Bluebonnet 50; CR, California Calrose; S, Louisiana Saturn; CL, California Caloro; CS, California Colusa; CB, California Belle Patna; CC, California Calrose; TE, Texas Belle Patna, early seeding; TL, Texas Belle Patna, late seeding; CP, California Pearl, parboiled, medium; TP, Texas Belle Patna, parboiled, light.

TABLE II  
Calcium and Magnesium Contents of Fractions of  
Overmilled Rice

Variety	Whole Kernel	Flour through 40-mesh Screen			Residual Kernel after Third Pass
		First Pass	Second Pass	Third Pass	
Calcium, mg. per 100 g. dry basis					
BP	38.5	343.2	140.2	94.3	27.2
BB	29.7	240.7	99.9	70.4	21.2
CR	25.1	177.5	78.7	55.2	18.1
S	18.4	100.5	46.9	44.0	13.1
CL	17.7	71.8	37.5	27.8	11.4
CS	18.1	71.8	37.6	29.5	12.0
Avg.	25	168	74	54	17
Std. dev.	± 8	±109	± 41	± 26	± 6
Magnesium, mg. per 100 g. dry basis					
BP	70.4	589	465	339	17.8
BB	20.1	316	233	128	5.1
CR	18.2	228	109	61	3.7
S	21.2	209	154	65	6.2
CL	17.1	257	128	74	6.5
CS	19.6	181	96	71	9.7
Avg.	28	297	198	123	8
Std. dev.	±21	±150	±140	±109	± 5

was compared with the amount in the original rice. The milled fractions analyzed—that is, the three flours through 40-mesh and the residual kernel—comprised  $95\% \pm 4\%$  (average and standard deviation) of the original rice. These samples accounted for 101% of the iron of the whole kernel rice, 86% of the calcium, 78% of the magnesium, 96% of the phosphorus, 71% of the sodium, 84% of the potassium, and 97% of the phytic acid. Differences from 95% may be due to variation in distribution of the component in the unanalyzed fractions and to experimental errors which involve the sum of analytical variations encountered in the determination of the weights of the fractions as well as the percent composition of the components. Residual kernels in particular were difficult to analyze accurately due to the small amounts of some of the constituents.

For each of the components studied, the values in the milled fractions were highest in the first-pass flours, decreased in all cases from the periphery to the interior of the kernel, and were lowest in the residual kernel. Of all the values for a given component including the various samples from all lots of rice, there was only a 5-fold difference in sodium content between the highest and the lowest values. But for potassium and phosphorus there was a 20-fold difference; for calcium, 30-fold; for magnesium, 160-fold; and for iron and phytic acid, more than 200-fold.

The average iron content of the original kernels for the 12 rices was 1.04 mg. per 100 g. of sample, and that of the residual kernels, 0.32 mg. (Table I). Flours averaged 15, 8, and 6 mg. for the first, second, and third passes, respectively. The average iron content of the first-pass flours was 14 times as high as that of the whole kernel, while residual kernels contained only about 30% as much iron as was found in the original rice.

The highest iron contents were found in the five lots of Belle Patna; Pearl and the two Calrose rices were intermediate; Bluebonnet 50, Saturn, and Colusa were lowest. Values for the flours of Caloro were anomalous. Although the iron contents of the original and residual kernels of CL were the lowest of the 12 rices studied, the flours had unreasonably high values. Calculation of the amount of iron in the three flours accounted for 73, 65, and 38%, respectively, or 176% of that of the whole kernel, while the residual kernel accounted for 46% making a total of 222%. The lot of rice, CS, had a somewhat larger amount of iron in the original and residual kernels; but the three flours had lower values than did those of CL and accounted for only 22, 19, and 16% of the iron in the original rice with the residual kernel 40%, for a total of 97%. The determinations on CL were repeated several times—a total of nine replicates for the whole kernel from two separate samplings of the total batch of whole kernel rice, and six replicates each for the second-pass flour and residual kernel. Precision of all determinations was good. Because CL was the first lot of experimental rice to be milled on the new mill, it was considered possible that traces of iron might have contaminated the milled fractions. Difficulty had been encountered with CL in determining ash content because particles of the millstone were found in the flour. Some of the stones were analyzed but no traces of iron were found, so that the abrasive cone itself was probably not the cause of the contamination.

The calcium content of six rice varieties averaged 25 mg. per 100 g. and that of the residual kernels, 17 mg. (Table II). Flours had values of 168, 74, and 54 mg. for the first, second, and third passes, respectively. Calcium content was highest

TABLE III  
Phytic Acid and Phosphorus Contents of Fractions  
of Overmilled Rice

Variety	Whole Kernel	Flour through 40-mesh Screen			Residual Kernel after Third Pass
		First Pass	Second Pass	Third Pass	
<b>Phytic Acid, mg. per 100 g. dry basis</b>					
BP	131	4,030	2,280	1,650	0
BB	120	3,570	1,850	1,180	0
CR	145	3,050	2,090	920	0
S	133	3,170	1,720	1,020	0
CL	134	1,910	1,120	660	0
CS	123	2,100	1,090	820	0
Avg.	132	2,972	1,690	1,040	0
Std. dev.	± 17	± 830	± 490	± 350	
<b>Phosphorus, mg. per 100 g. dry basis</b>					
BP	143	1,092	830	645	78
BB	146	1,200	862	458	113
CR	141	1,534	723	432	73
S	137	1,235	667	431	69
CL	86	845	521	278	73
CS	101	712	416	289	78
Avg.	126	1,103	670	422	81
Std. dev.	± 26	± 294	± 174	± 134	± 16

TABLE IV  
Sodium and Potassium Contents of Fractions of  
Overmilled Rice

Variety	Whole Kernel	Flour through 40-mesh Screen			Residual Kernel after Third Pass
		First Pass	Second Pass	Third Pass	
<b>Sodium, mg. per 100 g. dry basis</b>					
BP	8.6	14.3	10.9	9.3	6.3
BB	9.3	13.0	11.1	8.8	4.6
CR	10.0	13.0	11.7	8.6	6.1
S	10.0	17.9	13.5	10.5	9.3
CL	5.9	7.8	6.7	3.9	3.7
CS	4.8	6.9	6.5	5.3	4.4
Avg.	8.1	12.2	10.1	7.7	5.7
Std. dev.	± 2.2	± 4.1	± 2.8	± 2.6	± 2.0
<b>Potassium, mg. per 100 g. dry basis</b>					
BP	99	722	542	458	61
BB	91	747	399	258	55
CR	183	1,151	565	344	80
S	102	786	469	279	57
CL	142	600	374	253	108
CS	148	575	359	265	120
Avg.	128	764	451	310	80
Std. dev.	± 36	± 207	± 88	± 80	± 28

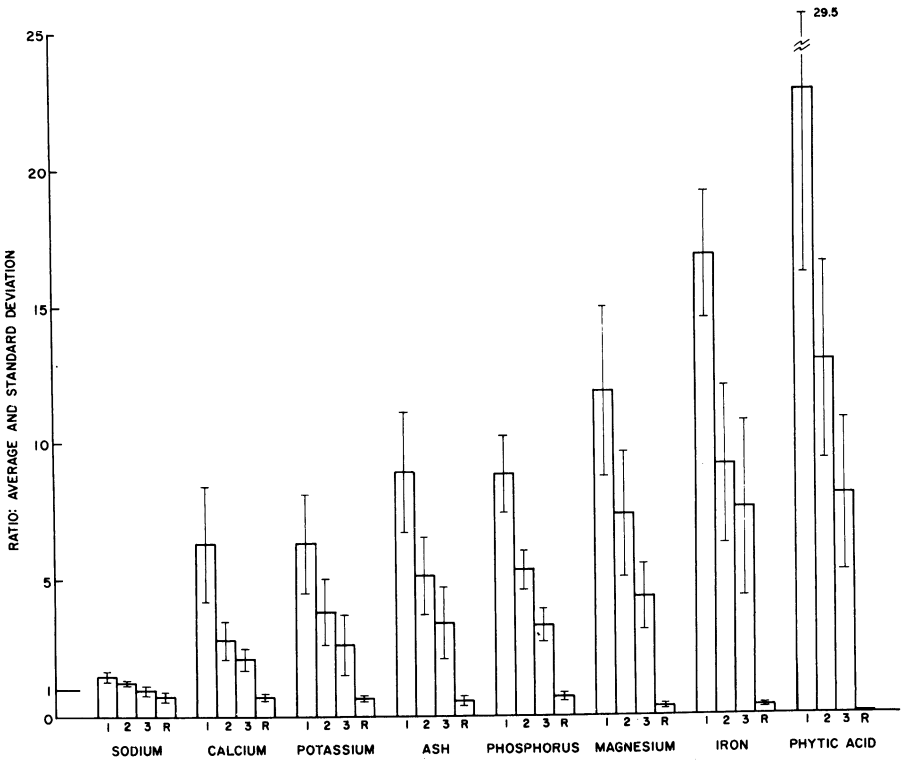


Fig. 1. Ratio and standard deviation of contents of sodium, calcium, potassium, ash, phosphorus, magnesium, iron, and phytic acid in flours and residual kernel of six varieties of rice, with respect to those of the original whole-kernel rice: 1, first-pass flour; 2, second-pass flour; 3, third-pass flour; R, residual kernel. For iron,  $n = 5$ , Caloro excluded.

in the samples of long-grain BP while little difference was shown in the values for corresponding fractions of CL and CS, short-grain rices with the lowest contents. Calcium values for the residual kernels averaged about 70% that of the whole kernels.

Magnesium contents of the whole and residual kernels averaged, per 100 g. sample, 28 and 8 mg., respectively, and the flours 297, 198, and 123 mg. (Table II). Values for BP were considerably higher than those for the other varieties. Although the magnesium and calcium values of the original rices were about the same, the flours had twice as much magnesium as calcium and the residual kernels less than half as much. Magnesium contents in the residual kernels averaged 30% that of the original rices.

Average phosphorus contents for whole and for residual kernels were 126 and 81 mg. per 100 g., respectively (Table III). The flours had averages of 1103, 670, and 422 mg. for the first, second, and third passes. Except for the residual kernels, fractions of the two short-grain rices consistently gave the lowest values. Phosphorus in the residual kernels averaged 66% of that in the whole kernels.

Sodium in the whole kernels averaged 8.1 mg. per 100 g. for the original rice and 5.7 mg. for the residual kernels, about 72% of that of the whole kernel (Table

TABLE V  
Summary of Mineral Components in Milled Samples from Six Varieties of  
Overmilled Rice, Average and Standard Deviation, 1966 Crop

Components	Whole Kernel	Flour through 40-mesh Sieve			Residual Kernel after Third Pass
		First Pass	Second Pass	Third Pass	
<b>Content in Samples, dry basis</b>					
Phytic acid, %	0.13 ± 0.01	2.97 ± 0.83	1.69 ± 0.49	1.04 ± 0.35	0
Ash, % <sup>a</sup>	0.60 ± 0.14	5.5 ± 2.7	3.2 ± 1.6	2.1 ± 1.4	0.31 ± 0.05
P, mg./100 g.	126 ± 26	1,103 ± 294	670 ± 174	422 ± 134	81 ± 16
K, mg./100 g.	128 ± 36	764 ± 207	451 ± 88	310 ± 80	80 ± 28
Mg, mg./100 g.	28 ± 21	297 ± 150	198 ± 140	123 ± 109	8 ± 5
Ca, mg./100 g.	25 ± 8	168 ± 109	74 ± 41	54 ± 26	17 ± 6
Na, mg./100 g.	8.1 ± 2.2	12.2 ± 4.1	10.1 ± 2.8	7.7 ± 2.6	5.7 ± 2.0
Fe, mg./100 g.	0.62 ± 0.45	11.5 ± 6.4	6.8 ± 5.4	5.4 ± 4.6	0.19 ± 0.09
<b>% Ash</b>					
P	21.4 ± 4.0	22.0 ± 5.9	22.8 ± 5.2	22.2 ± 4.9	26.6 ± 8.1
K	22.3 ± 7.7	15.4 ± 4.3	15.8 ± 4.2	16.7 ± 4.3	25.1 ± 5.0
Mg	4.3 ± 2.0	5.5 ± 1.4	5.9 ± 1.5	5.5 ± 1.6	2.6 ± 1.6
Ca	4.1 ± 0.8	2.9 ± 1.1	2.3 ± 0.6	2.7 ± 0.8	5.7 ± 2.4
Na	1.37 ± 0.36	0.24 ± 0.08	0.35 ± 0.11	0.42 ± 0.16	1.94 ± 0.93
Fe	0.10 ± 0.05	0.21 ± 0.07	0.20 ± 0.09	0.24 ± 0.10	0.06 ± 0.03
Total	53.6 ± 6.1	46.2 ± 9.9	47.3 ± 8.0	47.7 ± 8.3	62.0 ± 6.7
<b>% in Fraction based on Whole Kernel, dry basis</b>					
Phytic acid	100	48.5 ± 18.8	30.8 ± 8.2	17.6 ± 5.8	0
Ash	100	19.1 ± 7.7	12.2 ± 2.8	7.4 ± 2.7	48.6 ± 15.9
Phosphorus	100	18.6 ± 6.3	11.6 ± 3.1	7.4 ± 1.9	58.3 ± 14.3
Potassium	100	13.5 ± 5.5	9.0 ± 3.0	5.7 ± 2.3	55.7 ± 12.7
Magnesium	100	23.9 ± 7.1	17.1 ± 2.9	9.1 ± 1.2	27.6 ± 9.6
Calcium	100	13.5 ± 5.9	6.7 ± 1.4	4.7 ± 1.4	61.1 ± 3.6
Sodium	100	3.1 ± 0.9	3.0 ± 0.7	2.2 ± 0.8	63.0 ± 14.2
Iron	100	35.9 ± 11.0	21.4 ± 2.9	15.3 ± 3.1	28.4 ± 7.1
Milled fraction % of original rice, dry basis	100	2.1 ± 0.4	2.4 ± 0.4	2.3 ± 0.6	88.1 ± 3.9

<sup>a</sup>Ref. 4.

IV). First-, second-, and third-pass flours had averages of 12, 10, and 8 mg., respectively. Although the amount of sodium increased in the first-pass flours, the increase was only half again as much as in the whole kernel, the smallest of any of the components studied.

Potassium content of the whole kernels of the six varieties averaged 128 mg. per 100 g. (Table IV). Values were again higher in the flours, averaging 764, 451, and 310 mg. for the first-, second-, and third-pass flours. Residual kernels gave an average of 80 mg., 63% of that found in the whole kernels.

Phytate, calculated as phytic acid, averaged 132 mg. in the whole kernels (Table III). The content was greatly increased in the first-pass flours, averaging 3.0%—23 (15 to 31) times as great as in the original rices. Average values of phytic acid in the second- and third-pass flours were 1.7 and 1.0%, respectively. The highest percentages in the flours were found in the long-grain varieties, and the lowest in the short-grains. No phytic acid was detected in any of the residual kernels. Phytate phosphorus accounted for about 30% ( $30 \pm 7$ ) of the total phosphorus in the original rices, from 60 to 80% ( $73 \pm 11$ ) in the flours from all three passes, and none of the total phosphorus of the residual kernels.

## DISCUSSION

The high concentrations of minerals and phytic acid in the flours from the outer layers of the endosperm as compared with concentrations in the original rices are shown in Fig. 1. Sodium was the most evenly distributed throughout the kernel, as indicated by the lowest ratios for the flours and the highest for the residual kernels, while phytic acid showed the greatest differences in distribution, having the highest ratios for the first-pass flours and the lowest for the residual kernels.

The flours through 40-mesh from the three milling passes, which made up about 6% of the original rice, accounted for nearly all of the phytate in the whole kernels; none could be detected in the residual kernels. These three flours also accounted for 73% of the iron, 50% of the magnesium, 38% of the ash (4) and phosphorus, 28% of the potassium, and 25% of the calcium, but only 8% of the sodium. The first-pass flours alone contained half of the phytic acid and one-third of the iron.

Varietal differences are apparent. Of the 12 rices analyzed for iron, the five lots of Belle Patna (BP, CB, TE, TL, and TP) had the highest concentrations in the original rices and in the first-pass flours. Of the six varieties, the Belle Patna BP had the highest concentrations of calcium and of magnesium for both flours and kernels, and the highest phytic acid concentrations in all of the three flours. In all fractions the short-grain varieties, CL and CS, had the lowest concentrations of total ash (4), calcium, phosphorus, and sodium and, except in the original kernel, of phytic acid.

The six mineral elements analyzed in this study accounted for from 45 to 60% of the total ash (Table V). They made up about 54% of the ash of the whole kernels, 47% that of the flours, and 62% that of the residual kernels. Phosphorus and potassium composed the bulk of the ash. When compared with the whole kernel, potassium, calcium, and sodium as percentage of the total ash were lower in the flours and higher in the residual kernels, while magnesium and iron were higher in the flours and lower in the residual kernels. Phosphorus values as



percentage of total ash showed no definite trend. Varietal differences with respect to ash were not pronounced for any of the minerals except that, for the long-grain variety BP, phosphorus as percentage of total ash was lower for the flours, 10 to 13%, and higher, 25%, in the residual kernels when compared with the whole kernels, 17%.

One ash constituent, silicon, may possibly account for several percent of the total ash, particularly in the flours. Silicon has been reported to be present in white rice, 5 to 8 mg. Si per 100 g. dry basis (11). Unless rendered insoluble, silica interferes with the determination of calcium. Although not determined quantitatively in this study, silica could be seen as a precipitate in our treated ashes from the flours whereas little if any was noticed in ashes of the residual kernels. More precipitate was apparent in ashes of the long-grain rices than in those of the short-grains.

Manganese, zinc, and copper, none of which was analyzed in the present study, would be less significant with respect to total ash, since reported values for these elements in milled rice are 3 mg. or less per 100 g. (12).

Data for phosphorus and calcium for our sample of Bluebonnet 50 are similar to those of Normand et al. (3) for flours abraded from a sample of commercially milled, long-grain rice, predominantly Bluebonnet 50, by means of an experimental laboratory mill. In both studies the phosphorus contents of the first-pass flours were 8 to 9 times as great as for the original rice, and the flours from the outer kernel (4.8% in our study, 6.3% in Normand's) accounted for about 30 to 40% of the phosphorus in the original rice. Normand et al. reported only 28 mg. phosphorus per 100 g. residual kernel, however, whereas we obtained 113 mg. as compared with 140 and 146 mg., respectively, for the original rices. For calcium in the same lots of rice, Normand et al. reported a 20-fold increase in the first-pass flour with 85% of the calcium of the whole kernel accounted for in the flours, whereas we found only an 8-fold increase with 24% of the calcium accounted for. The Normand study gave no value for calcium in the residual kernel while we obtained 21 mg. per 100 g. as compared with 23 and 30 mg., respectively, in the whole kernel. There was a much steeper gradient in the rice studied by Normand et al., as shown by the sixth-pass flour after removal of the outer 11% of the kernel, which contained 16 mg. calcium per 100 g. flour, and the seventh-pass, which contained only 1 mg.

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