The Mineral Composition of Triticales and Triticale Milling Fractions by X-Ray Fluorescence and Atomic Absorption

K. LORENZ,[1] F. W. REUTER,[2] and C. SIZER[1]

ABSTRACT

Five winter and spring varieties of Triticale were milled on a Quadrumat Senior mill. The whole grain, flour, bran, and shorts of each of the varieties were analyzed for mineral elements by energy-dispersive X-ray fluorescence and atomic absorption. The precision of the results of X-ray fluorescence was shown to be comparable to that obtained with other methods of analysis. The main mineral elements of Triticale were shown to be potassium, phosphorus, magnesium, and calcium. Generally, bran contained the highest amounts of calcium, potassium, magnesium, sodium, manganese, iron, copper, and zinc, and flour contained the lowest amounts of those minerals. Shorts had intermediate amounts of those nutritionally important mineral elements. Comparing the mineral composition of Triticales with that of wheats showed Triticales to be higher in potassium, phosphorus, magnesium, sodium, manganese, iron, copper, and zinc. Triticale proved to be a good source of iron. Bromine, which is not detectable by atomic absorption and is usually not analyzed for by other chemical methods, was readily detectable by X-ray fluorescence. It was concluded that Triticale grains and flours, as well as the feed fractions—bran and shorts—could serve as a good source of mineral nutrients.

Triticale is a man-made cereal grain which has found increased use not only as an animal feed (1,2,3), but also for the production of many food products (4,5,6). Cereal grains, flours, and millfeeds contain many minerals essential for proper nutrition of man and animals. The mineral compositions of the parental species of Triticale—wheat and rye—have been reported (7-11). Data on the mineral composition of Triticales are lacking. In view of the potential value of Triticales as a food for human consumption, a comparison of the nutritive value of this man-made cereal grain with that of its parental species seems essential.

The objectives of this study were twofold. The first was to determine mineral composition in samples of Triticale and to assess the use of energy-dispersive X-ray fluorescence spectroscopy as a method of nondestructive mineral determination. Previous investigations of the mineral composition of cereals (7-10) used an ashing procedure followed by atomic absorption spectroscopy or various separation steps before measurements by colorimetric, flame-photometric, and titration techniques could be conducted. These analyses destroy the sample. X-ray fluorescence spectroscopy, however, is nondestructive (enabling samples to be reanalyzed in case of argument), requires little or no sample preparation (in particular, solids and powders need not be dissolved), and is sensitive enough to perform quantitative trace elements analysis. Energy-dispersive X-ray fluorescence spectroscopy has an additional advantage of simultaneous multielement analysis (12).

The second objective was to determine the changes in mineral distribution caused by milling of the grains into flour, shorts, and bran and to compare the

[1]Respectively: Associate Professor and Graduate Research Assistant, Department of Food Science and Nutrition, Colorado State University, Fort Collins 80521.

[2]Nuclear Engineer, Chemical Analysis Research, USDA, ARS, Western Regional Research Laboratory, Berkeley, Calif. 94710.

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mineral contents of Triticales and Triticale milling fractions with those of the parental species of Triticale as reported in the literature.

MATERIALS AND METHODS

Sample Identification

Samples of two spring and two winter Triticales, grown on irrigated sites in Colorado, as well as a commercial winter Triticale variety, were studied. The two spring Triticales (6-TA-204 and 6-TA-206), the two winter Triticales (TR-385 and TR-386), and the commercial sample WT-66 were milled on a Quadrumat Senior mill. The Triticale samples were tempered 18 hr. to 15% moisture. The milling data and the protein analyses of the grains and the flours as determined by AACC methods (13) are given in Table I.

X-Ray Fluorescence Analysis

The trace mineral composition of the Triticales and Triticale mill fractions was measured on a Finnigan Model 70 X-ray spectrometer. The principal components of the instrument are shown in Fig. 1.

Rhodium X-rays are generated by 40 kv. electrons striking a rhodium target. The X-rays are collimated and the beam is filtered through 0.003 in. of rhodium foil. K and L-shell fluorescence X-rays are excited in the sample. The beam size at the sample is about 1 $cm.^2$ The fluorescence radiation of the trace elements is collimated and measured in a lithium-drifted silicon detector. Each X-ray results in an electronic pulse proportional to the incident X-ray energy. The pulses are stored according to energy in a multichannel analyzer and the full spectrum is displayed on an oscilloscope screen. Data are usually collected for 1,000 sec. Low-concentration samples such as flour are run overnight.

The calibration and quantitation of X-ray peak areas to mineral content in the sample are patterned after the method of Giauque et al. (14, 15). A 300-mg. sample is pressed into a flat disc 1.25 in. in diameter and 1 mm. thick. The thin sample enhances the signal to noise ratio, and to first order makes the peak area proportional to the mineral content. The instrument is calibrated once with standard element concentrations in cellulose to determine the constant of proportionality between the peak area and elemental concentration. Deviations

TABLE I. MILLING DATA AND PROTEIN ANALYSES OF TRITICALES

Cultivar	Reduction Flour %	Break Flour %	Total Flour %	Shorts %	Bran %	Grain Protein[a] %	Flour Protein[a] %	Flour Amylograph Viscosity B.U.
Spring Triticales								
6-TA-204	29.1	31.9	61.0	7.2	31.8	15.7	12.7	690
6-TA-206	36.4	26.4	62.8	6.9	30.3	15.2	12.8	310
Winter Triticales								
TR-385	42.1	20.8	62.9	8.1	29.0	13.1	11.6	680
TR-386	44.3	24.0	68.3	7.0	24.7	13.5	11.5	840
WT-66	29.7	26.8	56.5	6.9	36.6	12.8	10.2	720

[a]14% moisture basis.

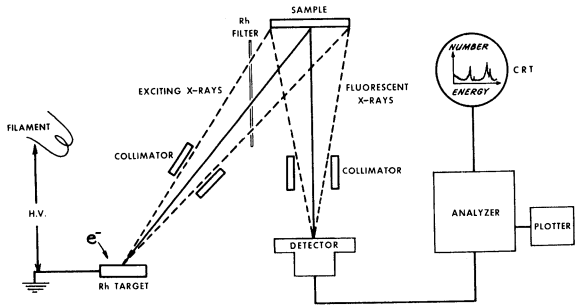

Fig. 1. X-ray fluorescence spectrometer.

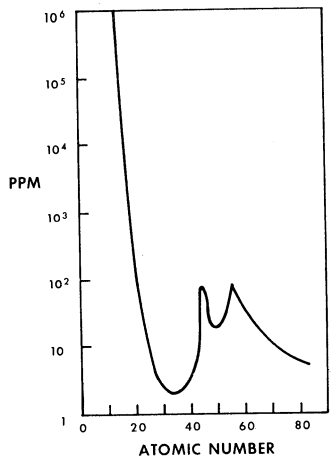

Fig. 2. X-ray detection limits in biological material.

from linearity between concentration and X-ray signal are corrected by a matrix absorption factor which measures X-ray absorption in the sample. This factor is 60% or less for trace element determinations and is constant for similar samples. The basic technique will accurately measure concentrations as high as 50%.

The principal advantages of the technique allow one to simultaneously and nondestructively determine the elements present in a sample without any prior knowledge of them. A curve of the detection limits in biological material is shown in Fig. 2. The curve was prepared with the appropriate conversion factors for each element assuming an elemental peak area three times the standard deviation of the background signal after data collection for 1,000 sec. The two humps in the curve result from the exciting X-ray energy and the X-ray energy range detected. They can be shifted by changing these two parameters. Rhodium K-shell X-rays were chosen as the exciting radiation to yield the maximum number of trace elements and pollutants commonly found in biological material. The results of a recent measurement of National Bureau of Standards, Orchard Leaves, Standard Reference Material No. 1571, gave the results shown in Table II. Numbers preceded by a less

TABLE II. TRACE ELEMENT CONCENTRATION IN N.B.S. ORCHARD LEAVES

Element	Concentration, p.p.m.	
	X-ray Fluorescence	N.B.S.
Cr	< 18.3[a]	2.3[b]
Mn	101.0 ± 10	91.0 ± 4
Fe	319.0 ± 32	300.0 ± 20
Co	< 5.7[a]	0.2[b]
Ni	< 3.9[a]	1.3 ± 0.2
Cu	13.8 ± 1.4	12.0 ± 1
Zn	27.4 ± 2.7	25.0 ± 3
As	12.3 ± 1.2	14.0 ± 2
Br	8.9 ± 0.9	10[b]
Rb	11.5 ± 1.2	12.0 ± 1
Sr	42.2 ± 4.2	37[b]
Pb	47.1 ± 4.7	45.0 ± 3

[a]Not detected; concentration less than the detection limit shown.
[b]No standard deviations reported.

TABLE III. ATOMIC ABSORPTION OPERATING CONDITIONS AND INSTRUMENTAL PARAMETERS

Element	Cathode Lamp. Mamp. Current	Wavelength Å	Air p.s.i.	Acetylene p.s.i.	Analytical Range p.p.m.
Na	10	5,890	9.0	9.0	0.3-3
Mg	6	2,852	8.5	9.0	0.1-2
K	12	7,665	9.0	9.0	1-10
Ca	10	4,227	7.5	9.0	1-10

than sign were not detected. The concentrations shown are taken from the detection limits in Fig. 2. The accuracy of measurement is ±10%. The principal disadvantage of this method of mineral analysis is the detection limit, which does not permit the determination of all the nutritionally important mineral elements.

Atomic Absorption Spectroscopy

Ashing Procedure. For the analyses of Na, Mg, K, and Ca which could not be determined by X-ray fluorescence, approximately 4-g. samples of the ground whole grain, the flour, bran, and shorts of each of the Triticale samples were digested in 250-ml. Erlenmeyer flasks with 10 ml. HNO_3 and 0.5 ml. of a concentrated $HClO_4$-H_2SO_4 (1:9) mixture. The samples were heated gently on a hot plate to avoid foaming. Heating was continued until solutions were clear. Solutions, which did not become clear, were allowed to cool. An additional 10 ml. of HNO_3 was added and heating was continued until the contents of the flasks were clear and almost dry. The solutions were allowed to cool after which 2 ml. of concentrated HNO_3 were added to the flasks which then were heated gently with a cover glass in place to get a reflux action. The walls of the flasks were rinsed with distilled water and the mixture evaporated to 2 ml. Approximately 10 ml. of distilled water was added and the total contents transferred to 25-ml. volumetric flasks which were made up to volume.

TABLE IV. MINERAL COMPOSITION OF TRITICALES AND TRITICALE MILLING FRACTIONS (dry basis)[a]

	Ash %	K %	Mg %	Ca %	P %	Na p.p.m.	Mn p.p.m.	Fe p.p.m.	Cu p.p.m.	Zn p.p.m.	Br p.p.m.	Rb p.p.m.	Sr p.p.m.
Grain													
WT-66	2.30	0.358	0.191	0.027	0.503	39	51.7	55.4	4.3	35.9	19.5	< 2.7	< 2.9
6-TA-204	1.76	0.430	0.185	0.033	0.465	55	57.4	44.7	9.8	24.4	34.0	6.3	3.3
6-TA-206	2.28	0.508	0.198	0.035	0.494	43	63.2	46.4	< 3.2	27.6	28.6	< 2.7	7.0
TR-385	1.98	0.459	0.191	0.032	0.437	29	48.6	57.0	13.8	24.5	50.4	< 2.7	9.1
TR-386	1.93	0.432	0.184	0.036	0.534	57	56.0	54.0	5.3	18.0	30.6	< 2.7	3.4
Mean	2.05	0.437	0.190	0.033	0.487	45	55.4	51.5	7.3	26.1	32.6	3.4	5.1
Flour													
WT-66	0.52	0.144	0.031	0.014	0.174	32	< 12.9	16.5	< 3.2	4.8	20.0	< 2.7	< 2.9
6-TA-204	0.53	0.176	0.030	0.018	0.173	46	< 12.9	8.8	< 3.2	3.6	28.7	< 2.7	< 2.9
6-TA-206	0.63	0.184	0.030	0.029	0.190	45	< 12.9	10.0	< 3.2	5.0	23.8	< 2.7	< 2.9
TR-385	0.53	0.149	0.034	0.013	0.191	30	< 12.9	14.8	< 3.2	2.9	25.4	< 2.7	< 2.9
TR-386	0.51	0.165	0.041	0.035	0.216	33	< 12.9	13.4	< 3.2	4.2	24.2	< 2.7	< 2.9
Mean	0.54	0.164	0.033	0.022	0.189	37	< 12.9	12.7	< 3.2	4.1	24.4	< 2.7	< 2.9
Bran													
WT-66	4.18	0.972	0.652	0.040	0.266	52	126	128	11.6	79.4	25.7	5.2	7.9
6-TA-204	3.83	1.064	0.723	0.054	0.276	82	125	113	14.1	59.8	44.0	11.2	12.2
6-TA-206	3.99	1.077	0.521	0.061	0.293	60	157	129	2.6	72.9	35.5	9.4	11.5
TR-385	4.26	0.840	0.775	0.063	0.462	58	178	184	3.1	50.3	33.4	4.3	10.2
TR-386	4.21	1.003	0.422	0.049	0.373	42	182	164	15.6	57.4	37.3	5.5	15.4
Mean	4.09	0.991	0.619	0.053	0.334	59	154	144	14.2	64.0	34.2	7.1	12.4
Shorts													
WT-66	2.03	0.382	0.160	0.030	0.577	58	48.3	45.4	7.0	26.2	21.1	5.2	< 2.9
6-TA-204	1.83	0.455	0.164	0.039	0.348	52	86.8	53.9	7.4	28.9	27.9	6.0	11.1
6-TA-206	1.99	0.369	0.159	0.036	0.442	40	86.7	53.7	8.4	35.5	27.7	4.1	5.4
TR-385	2.00	0.437	0.199	0.039	0.649	53	72.9	88.2	9.7	20.9	26.1	< 2.7	4.9
TR-386	2.18	0.458	0.203	0.042	0.436	41	79.7	76.2	9.9	22.4	29.7	3.1	8.7
Mean	2.01	0.420	0.177	0.037	0.490	49	74.9	63.7	8.5	27.0	26.5	4.2	6.6

[a]The amounts of Mn, Cu, Rb, and Sr are given as < for certain milling fractions.

Instrumentation. A Perkin-Elmer Model 303 atomic absorption spectrophotometer was used and operated for absorption measurements as shown in Table III. Single element hollow cathode lamps were used for the determinations at the recommended current rating. A set of standards was run at the beginning and at the end of each series of samples. The primary standard stock solutions were prepared from chemicals of high purity. Secondary working standard solutions were prepared from the primary stock solutions covering the desired concentration range for each element. Two nonconsecutive readings were recorded for samples and background. An aliquot of the sample solutions was diluted as required to give an optimum concentration for the absorbance readings.

No detectable interferences were found in the determinations of Na, Mg, and K. The interference of phosphorus in the determination of Ca was eliminated by using the procedure as described by Garcia et al. (16).

Phosphorus Determination. Phosphorus was determined according to AACC Method 40-55 (13).

RESULTS AND DISCUSSION

Milling Results

The milling data in Table I indicate that the flour extractions for the Triticale samples are considerably lower than those normally obtained with wheat samples. The lower extraction rates are due to the shrivelled condition of the Triticale kernels, which is one of the serious shortcomings of this man-made cereal grain (17). The amount of bran produced is considerably higher in Triticale milling than in milling of wheat. The percentage of Triticale shorts is comparable to the percent shorts reported in the milling of wheat varieties (18). The lower extraction rates found in this study agree with those reported by others (4,19,20).

Mineral Composition of Triticales and Triticale Milling Fractions

The contribution of a cereal grain to the minerals in the diet depends on the milling fraction. The minerals are not uniformly distributed in wheat and rye and the same is true for Triticales.

TABLE V. MINERAL ELEMENT CONCENTRATION IN TRITICLES COMPARED TO AMOUNTS IN WHEATS AND RYE

Element	Soft Wheats[a]	Hard Wheats[a]	Durum Wheats[a]	Rye[b]	Triticales
K (%)	0.418	0.414	0.494	0.52	0.437
P (%)	0.340	0.344	0.370	0.38	0.487
Mg (%)	0.159	0.180	0.186	0.13	0.190
Ca (%)	0.039	0.037	0.034	0.07	0.033
Na (p.p.m.)	30	30	50	20	45
Mn (p.p.m.)	35	38	32	75.2	55.4
Fe (p.p.m.)	37	44	40	100	51.5
Cu (p.p.m.)	4.5	5.1	4.8	8.8	7.3
Zn (p.p.m.)	5	24	30	34.3	26.1
Se (p.p.m.)	0.28	0.50	0.69	---	---
Rb (p.p.m.)	---	---	---	---	3.4
Sr (p.p.m.)	0.48[c]	0.72[c]	---	---	5.1

[a]Tcepfer et al. (ref. 10).
[b]National Research Council, Committee on Animal Nutrition (ref. 11).
[c]Waggle et al. (ref. 8).

Table IV indicates variations among the Triticales in their contents of the 12 mineral elements analyzed. Data for mineral elements presented by Toepfer et al. (10) showed potassium, phosphorus, magnesium, and calcium, in decreasing order, to be the main mineral elements of whole wheat grain. Occasionally, however, phosphorus is the predominant mineral element as shown by Waggle et al. (8). The same five minerals were the main elements in Triticales.

Variations in concentration of mineral elements have been attributed to the effects of soil, conditions of growth, water supply, time of sowing, fertilizers, as well as varietal differences (21,22). The phosphorus content of wheat receiving irrigation tended to increase in experiments of Greaves and Hirst (23). The Triticale varieties investigated in this study were grown on irrigated sites which could explain the high phosphorus content of these samples. In three of the five samples (WT-66, 6-TA-204, and TR-386) phosphorus was the predominant mineral element followed by potassium, while in the other two samples (6-TA-206 and TR-385) potassium was the main mineral followed by phosphorus.

The amounts of the various mineral elements in the Triticale grain samples does not appear to be related to total ash content which was also the conclusion drawn after analyses of wheat samples (24). The concentrations of all elements were lower in the flours than in the grains.

The mineral elements were not present in the Triticale flours, the brans, and the shorts in proportions of the concentration in the grains. In general, bran contained the highest percentages of calcium, potassium, magnesium, sodium, manganese, iron, copper, and zinc; and flour contained the lowest percentages of these minerals. Shorts contained intermediate amounts of these nutritionally important mineral elements. This agrees with the general mineral distribution reported for wheats (7,8).

Specifically, only about 18% of the magnesium of Triticale grain was found in flour. Potassium, phosphorus, and iron were found in the flour to the extent of 25 to 40% and calcium and sodium to the extent of 67 and 82%, respectively, of their amounts in Triticale grains.

With an increase of the ash content in the bran fraction, the concentration of each of the mineral elements increased, although not to the same extent. Bran, which had approximately eight times as much ash as the Triticale flours, contained about seven times more potassium, between 1.5 and 2.5 times more sodium and calcium, 12 times more iron and manganese, and about 17 to 19 times more zinc and magnesium.

Triticale proved to be a very good source of iron, which is deficient in the diets of several groups of people. Bromine was found in relatively high amounts in Triticales and Triticale milling fractions. The element cannot be measured by atomic absorption and, therefore, is rarely reported. There is rarely a biological material, however, which does not contain the element (25). It is naturally ubiquitous and is an automobile pollutant as well. Since bromine was found in the flour fraction which comes from the inside of the kernel, external contamination is unlikely. Measurements of airborne dust by X-ray fluorescence indicated definite contamination with automobile emissions, bromine occurring in similar amounts as lead. Lead, however, was not detected in the Triticales. This causes us to conclude that the bromine was absorbed naturally from the soil. The trace mineral strontium has been reported in samples of wheat (8), although in lower amounts than those found in the Triticales.

The results presented here suggest that Triticale grains and flours as well as the feed fractions–bran and shorts–which are produced in relatively high proportions from the Triticales, could serve as a good source of mineral elements.

Mineral Content of Triticales vs. That of Wheat and Rye

A comparison of the mineral composition of Triticales with that of the parental species–durum wheat and rye–indicates that Triticales contain higher amounts of the major mineral elements potassium, phosphorus, and magnesium, possibly inherited from the parental species. Varietal effects influence mineral composition of grains (24). Rye contains higher amounts of potassium and phosphorus than the wheats, and durum wheat is slightly higher in magnesium, as seen in Table V. The nutritionally important minor elements sodium, manganese, iron, copper, and zinc are present in higher amounts in Triticales than in soft or hard wheats. The levels of those minerals tend to be intermediate between those reported for the parental species durum wheat and rye.

Amounts of rubidium and strontium in durum wheat and rye could not be found in the literature for comparison. The higher levels of those elements in Triticales compared to soft and hard wheats could be due to the better method of analysis for these mineral elements.

Triticales appear to be a better source of essential minerals than soft or hard wheats.

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