IRON ENRICHMENT OF DRY-MILLED CORN PRODUCTS[1]

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ABSTRACT

Powdered iron, reduced either electrolytically or with hydrogen, can be added to common dry-milled corn products, such as grits or meal, at levels of 40 mg iron/lb with little or no segregation of iron. Accelerated storage of grits and meal enriched at this level resulted in no adverse odors or flavors after 56 days. At the same levels, stabilized ferrous sulfate added to these products does not segregate appreciably, but there is some deterioration of flavor and odor during storage. When iron levels were increased to 200 mg/lb product, each iron source gave results similar to these. Soy-fortified corn meal enriched with either 40 or 200 mg iron/lb, as reduced iron or stabilized ferrous sulfate, showed no appreciable migration of iron or degeneration of flavor or odor after storage.

Iron-deficient diets have been recognized as a significant nutritional problem in the United States. It mainly manifests itself as anemia in infants and in the female population (1–4). Much has been written about the problem and considerable controversy exists as to the manner in which the diet should be supplemented with iron, and what the source of iron should be. In 1972, an Ad Hoc Committee on Iron Enrichment of Wheat Flour and Baked Goods (5) concluded that no single iron source is ideally suited for the enrichment of all flour and flour products. They limited their report to providing information on commercial availability and functional characteristics of iron compounds with a minimal review of iron assimilability, since this latter subject was considered to be difficult and controversial, requiring special expertise. They also reported that reduced iron, because of its inertness and low cost, is the iron source of choice for flour and other products requiring extended shelf-life. Results of bioavailability studies on reduced iron vary widely, possibly because of an apparent inverse relationship between availability and particle size. [This has also been reported by Canadian workers (6).] Ferrous sulfate, because of its high assimilability and relatively low cost, is the iron salt of choice for bread and other baked products where long shelf-life is not a factor (5,7).

The question of human bioavailability of iron is a broad and complex one and, as pointed out by the Ad Hoc Committee, it needs resolution by further research. Numerous workers have studied the assimilation of various iron sources and are in general agreement that low-cost ferrous sulfate has high availability, while the availability of widely used ferric orthophosphate is quite low (8–10). It has also been reported by the above workers that the bioavailability of reduced iron was rated as mediocre, but more recent information relates availability of this iron to particle size (5–7), with the finer powdered iron being rather highly assimilable.

Wheat flour, corn grits, corn meal, and farina have been enriched with iron for many years. The level of addition has been at least 13 mg iron/lb (11). In 1973, the

[1]Presented at the 59th Annual Meeting, Montreal, Canada, Oct. 1974.
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Mention of firm names or trade products does not imply that they are endorsed or recommended by the U.S. Department of Agriculture over other firms or similar products not mentioned.

Food and Drug Administration (FDA) proposed that wheat-flour standards of identity be revised to increase the level of supplementation to 40 mg/lb (12). The FDA has also proposed changes in the standards of identity for enriched farina to include increasing the level of iron addition to 8.1 mg/oz or 130 mg/lb (13).

Studies have been reported and continue on the enrichment of flour, baked goods, and infant cereals with iron (14–17). These studies have been related to absorption phenomenon and response of added iron to baking characteristics. We have conducted an investigation to determine some segregation properties and storage stability characteristics of corn grits and corn meal when they were fortified with different iron sources at levels of 40 to 200 mg addition/lb.

MATERIALS AND METHODS

Materials

Coarse and fine corn grits, degerminated corn meal, and soy-fortified corn meal were obtained through commercial channels; all met USDA specifications CG-1 (18), CM-4 (19), and SFCM-1 (20), respectively. Particle-size distributions of the commodities are as follows:

Item	*Coarse Corn Grits*		*Fine Corn Grits*		*Degerminated Corn Meal*		*Soy-Fortified Corn Meal*	
	Minimum	*Maximum*	*Min.*	*Max.*	*Min.*	*Max.*	*Min.*	*Max.*
Percentage through U.S. standard sieve								
No. 14	75	...	75	...	...	...	...	...
18	...	30	30	...	...	...	...	...
20	...	...	...	...	99	...	99	...
25	...	10	...	20	90	...	91	...
45	...	...	...	...	30	...	40	...
80	...	...	...	...	...	20	...	32

Iron samples studied were from commercial sources and included powdered iron electrolytically reduced [reduced iron (E)]; powdered iron reduced with hydrogen [reduced iron (H)]; stabilized ferrous sulfate; and reduced iron (H), ferrous sulfate, or ferric orthophosphate in combination with thiamine, riboflavin, niacin, and starch carrier (will be referred to as "iron source—always named—in starch"). The following table lists the particle-size distribution of the iron sources:

Iron Source	*Particle Size*
Powdered iron, electrolytic	min. 95% through 325 mesh
Powdered iron, hydrogen	min. 95% through 325 mesh
Stabilized ferrous sulfate	99% through 100 mesh
Ferrous sulfate in starch	70–85% through 100 mesh 35–50% through 325 mesh
Reduced iron in starch	100% through 100 mesh 90–95% through 325 mesh
Ferric orthophosphate in starch	70–80% through 325 mesh

EXPERIMENTAL METHODS

Iron Segregation

Segregation tests were designed to simulate the movement of a container of material while being processed and transported. A commercial-scale test in which reduced iron (H) in starch was added to corn grits and iron migration traced from packaging line to final destination some 800 miles away indicated that more segregation occurred during the packaging than in transit. We then designed a test which we believe to be more drastic than ordinary lifting, moving, or bouncing of cartons in a railroad car or on a truck. In the first test, a shaker test, 200 g of enriched sample is placed in a jar, which is fastened to the platform of a Burrell wrist-action shaker, and the jar is gently shaken for 5 min at the rate of 340 cycles/min. The second test, a pounding test even more severe than the above test, involves placing the enriched sample in a jar, which is lifted and dropped about 1/2 in. onto a rubber pad by action of the shaker. The jar is lifted and dropped at the rate of 340 cycles/min for 5 min.

The different iron sources were added to the four dry-milled corn products at levels of approximately 40, 100, and 200 mg iron/lb of product by blending ingredients in a container fitted with baffles and rotated for 1 hr on a ball mill. After subjecting the enriched material to the shaking and pounding tests, samples were taken from the top and bottom of the jar and analyzed for iron. For the pounding test on material enriched with 100 mg of iron, samples were taken from the top, middle, and bottom of each jar.

Storage Stability Tests

Samples of the iron-enriched dry-milled corn products were subjected to accelerated storage tests; *i.e.*, 28 and 56 days' storage at 49° C, after which they were evaluated for color, odor, and flavor by a four-member expert panel. Samples stored in glass jars were given qualitative ratings in the uncooked state, for visual color comparison with a control and for odors, by comparing the aroma of the stored samples to corresponding fresh corn products. They were evaluated for flavor as cooked gruels, at 10% solids, made by stirring into boiling water and cooking for 1 min. The gruels were rated on the basis of the 10-point score sheet described by Bookwalter *et al.* (21). A score of 10 meant sample was excellent, 9 = very good, 8 = good, etc. Scores of 6 and above were considered acceptable. Objectionable flavors were assigned scores of 5 and below. The objective of the panel was to determine difference or flavor change of the stored samples when compared to a control containing no added iron.

Analytical and Statistical Methods

Iron was determined by dry-ashing 25 g of the enriched sample, dissolving ash in 2% hydrochloric acid, and measuring Fe in the solution by atomic absorption[3]. Standard deviations were estimated from the highest-order interactions as well as variation between duplicates.

EXPERIMENTAL RESULTS AND DISCUSSION

Segregation of Iron in Dry-Milled Corn Products

When iron or any ingredient is added to a cereal or cereal product, there always

[3]Private communication with L. T. Black, NRRL, Peoria, Ill.

exists the possibility of segregation or migration of the added material. This phenomenon is particularly likely when the particle size of the cereal fraction is rather coarse, as in a meal or in grits. The addition of powdered iron, either in the form of pure iron or as a salt, to these coarser products offers potential problems of segregation during manufacture and shipment of the final packaged material.

In Table I, results are given for segregation tests carried out by the shaker method involving the addition of four powdered iron sources to four dry-milled corn products at levels of 40 and 200 mg iron/lb of product.

A statistical analysis of the data from the 40-mg addition showed that segregation of iron from top to bottom or bottom to top (noted as "sample location") was not significant at the 5% level. There were also no significant interactions involving location and iron source or corn product. Statistical results were the same for samples enriched at the higher iron addition.

Coarse and fine grits fortified with several iron sources were subjected to the more severe test for segregation, the so-called "pounding test" described earlier in this paper. Three levels of iron were added—40, 100, and 200 mg iron/lb of grits (Table II).

At 40-mg iron addition, no significant effects were observed. There was a significant interaction of sample location and iron source at the 100-mg level. Segregation was high for the ferrous sulfate in starch, whereas it was low for the other sources tested.

At 200-mg iron enrichment, there was no significant effect with respect to sample location.

Standard deviations (S) for the 40- and 200-mg iron additions were:

Iron addition, mg	*S*	*LSD*[4] *between two values*
40	5.2	16
200	14.3	44

No segregation of protein or available lysine occurred in any of the corn products studied during either the shaker test or the pounding test.

Storage Stability of Iron-Enriched Dry-Milled Corn Products

Storage stability characteristics of iron-fortified corn products were evaluated after accelerated tests; *i.e.*, 28- and 56-day storage at 49°C. These short-term "stress" conditions are approximately equivalent to storage of corn-based materials for 6 months at 37°C or for 12 months at 25°C (22,23).

Evaluations were made at the extreme levels of iron additions; *i.e.*, 40 and 200 mg/lb, to the four corn products tested.

Color and Odor. Color and odor evaluations were made on uncooked products, comparing the stored iron-enriched product with a corresponding sample that had been maintained at −18°C for the same storage period.

Addition of the three different reduced irons at the 40 mg/lb level changed neither color nor odor in either coarse grits or corn meal after accelerated storage for as long as 56 days (Table III). No odor changes were noted after storing the fine grits and soy-fortified corn meal, but there was a slight dulling of the color.

The color of the corn products to which stabilized ferrous sulfate was added

[4]Least significant difference at 0.05 level.

TABLE I
Segregation of Iron in Iron-Enriched Dry-Milled Corn Products (Shaker Test[a]) (mg Iron/lb Cereal Product)

Corn Product	Iron Source							
	Reduced iron (E)		Reduced iron (H)		Reduced iron (H) in starch[b]		Stabilized $FeSO_4$	
	Top	Bottom	Top	Bottom	Top	Bottom	Top	Bottom
	Fortification—40 mg/lb							
Coarse grits	40	38	37	39	39	35	43	47
Fine grits	41	36	49	36	46	47	43	53
Degerminated corn meal	42	46	43	27	38	39	46	55
Soy-fortified corn meal	55	55	59	56	56	54	56	54
	Fortification—200 mg/lb							
Coarse grits	210	222	210	210	166	151	214	241
Fine grits	210	232	225	227	210	250	228	249
Degerminated corn meal	235	231	220	217	228	228	229	276
Soy-fortified corn meal	243	249	235	252	282	245	243	267

[a]Samples are taken from top and bottom of jars fastened to platform of a wrist-action shaker after shaking for 5 min.
[b]Iron in starch includes a combination of iron source, thiamine, riboflavin, niacin, and starch carrier.

TABLE II
Segregation of Iron in Iron-Enriched Corn Grits (Pounding Test[a]) (mg Iron/lb Cereal Product)

Iron Source	Coarse Grits			Fine Grits		
	Top	Middle	Bottom	Top	Middle	Bottom
	Fortification—40 mg/lb					
Reduced iron (E)	44	...	36	43	...	45
Reduced iron (H)	38	...	42	40	...	48
Reduced iron (H) in starch	38	...	44	52	...	42
Stabilized $FeSO_4$	45	...	47	54	...	38
	Fortification—100 mg/lb					
Reduced iron (H) in starch	120	115	156	133	136	134
$FeSO_4$ in starch	115	113	200	116	113	180
$Fe_3(HPO_4)_4$ in starch	122	124	127	132	121	132
	Fortification—200 mg/lb					
Reduced iron (E)	217	...	207	210	...	234
Reduced iron (H)	203	...	240	206	...	206
Reduced iron (H) in starch	178	...	207	241	...	256
Stabilized $FeSO_4$	240	...	226	250	...	256

[a]Samples are taken from top, middle, and bottom of jars which are lifted and dropped by a wrist-action shaker for 5 min.

TABLE III
Color and Odor of Uncooked Stored Dry-Milled Corn Products Enriched with 40 mg Iron/lb

	Stored at 49°C	Iron Source							
		Reduced iron (E)		Reduced iron (H)		Reduced iron (H) in starch		Stabilized $FeSO_4$	
Corn Product	Days	Color[a]	Odor[b]	Color	Odor	Color	Odor	Color	Odor
Coarse grits	28	100	Normal	100	Normal	100	Normal	100	Slightly rancid
	56	100	Normal	100	Normal	100	Normal	100	Rancid
Fine grits	28	99.5	Normal	99.5	Normal	99.5	Normal	99.5	Slightly rancid
	56	99.5	Normal	99.5	Normal	99.5	Normal	99.5	Rancid
Degerminated corn meal	28	100	Normal	100	Normal	100	Normal	100	Slightly rancid
	56	100	Normal	100	Normal	100	Normal	100	Rancid
Soy-fortified corn meal	28	99	Nutty	99	Nutty	99	Nutty	99	Nutty
	56	99	Nutty	99	Nutty	99	Nutty	99	Nutty

[a]Control: color = 100; 100 = light yellow; 99 = gray-yellow.
[b]Control: normal, defined as corn-like. For soy-fortified corn meal, nutty.

TABLE IV
Color and Odor of Uncooked Stored Dry-Milled Corn Products Enriched with 200 mg Iron/lb

		Iron Source							
	Stored at 49°C	Reduced iron (E)		Reduced iron (H)		Reduced iron (H) in starch		Stabilized $FeSO_4$	
Corn Product	Days	Color[a]	Odor[b]	Color	Odor	Color	Odor	Color	Odor
Coarse grits	28	99.5	Normal	99.5	Subnormal[c]	99.5	Normal	99.5	Slightly rancid
	56	99.5	Normal	99.5	Subnormal	99.5	Normal	99.5	Slightly rancid
Fine grits	28	99	Normal	99	Subnormal	99	Normal	99	Slightly rancid
	56	99	Normal	99	Subnormal	99	Normal	99	Slightly rancid
Degerminated corn meal	28	99	Normal	99	Normal	99	Normal	99	Slightly rancid
	56	99	Normal	99	Normal	99	Subnormal	99	Slightly rancid
Soy-fortified corn meal	28	99	Nutty	99	Nutty	99	Nutty	99	Nutty
	56	99	Nutty	99	Nutty	99	Nutty	99	Nutty

[a]Control: color = 100; 100 = light yellow; 99 = gray-yellow.
[b]Control: normal, defined as corn-like. For soy-fortified corn meal, nutty.
[c]Subnormal—not a rancid odor but less corn-like than control.

exhibited a similar change. However, a slightly rancid to rancid odor appeared in the enriched coarse and fine grits and in the corn meal. Soy-fortified corn meal enriched with this iron source had the same odor as the control.

In Table IV are the color and odor evaluations of corn products enriched with the four iron sources at the 200 mg iron/lb level. Color ratings of the four stored iron-enriched products were all slightly lower than the control. With the exception of the soy-fortified corn meal, color ratings were also less than those noted in the same products enriched with only 40 mg iron/lb. Odor was off slightly in the stored grits enriched with reduced iron (H) and at 56 days in corn meal that contained reduced iron (H) in starch. Grits and corn meal enriched with stabilized ferrous sulfate displayed slightly rancid odors. However, the soy-fortified corn meal had an odor comparable to the control. This phenomenon was also noted in soy-fortified corn meal enriched with the lower level of stabilized ferrous sulfate, and is thought to be due to naturally occurring antioxidants present in the soy flour component.

Flavor Stability. Flavor stability evaluated on the basis of cooked gruels prepared from the four stored corn products enriched with different iron sources at an addition level of 40 mg iron/lb is shown in Table V. Storage was for 28 and 56 days at 49°C. Average flavor scores changed slightly during storage for the corn products enriched at this level, decreasing as storage time progressed. Scores of samples of corn grits (coarse and fine) and corn meal enriched with stabilized ferrous sulfate decreased significantly with time, and were significantly lower than the same products enriched with the other iron sources. However, scores of soy-fortified corn meal enriched with stabilized ferrous sulfate did not change on storage.

Similar flavor stability tests were carried out on the corn products enriched with the four iron sources added at the level of 200 mg iron/lb (Table VI).

There was a significant reduction in flavor scores in all products enriched with the reduced iron sources as storage went from 0 to 56 days. Effects were not as significant, possibly reflecting lower flavor scores for the corn grits and meal at 0 days at the 200-mg level than were observed at 40 mg (Table V). Again, the coarse

TABLE V
Flavor Scores of Cooked Gruels Made from Stored Dry-Milled Corn Products Enriched with 40 mg Iron/lb

	Iron Source											
	Reduced iron (E)[a] days			Reduced iron (H)[a] days			Reduced iron (H) in starch[a] days			Stabilized $FeSO_4$[a] days		
Corn Product	0	28	56	0	28	56	0	28	56	0	28	56
Coarse grits	8.0	7.3	7.9	8.0	7.5	7.3	8.0	7.7	6.9	8.0	4.5	4.0
Fine grits	8.0	7.7	7.2	8.0	7.6	7.3	8.0	7.9	7.3	8.0	4.8	4.0
Degerminated corn meal	8.0	7.7	6.9	8.0	7.6	6.8	8.0	7.7	7.3	8.0	4.7	4.3
Soy-fortified corn meal	7.0	6.7	6.9	7.0	6.6	6.8	7.0	6.9	6.9	7.0	7.0	6.4

[a]Stored at 49°C.

TABLE VI
Flavor Scores of Cooked Gruels Made from Stored Dry-Milled Corn Products Enriched with 200 mg Iron/lb

	Iron Source											
	Reduced iron (E)[a] days			Reduced iron (H)[a] days			Reduced iron (H) in starch[a] days			Stabilized $FeSO_4$[a] days		
Corn Product	**0**	**28**	**56**	**0**	**28**	**56**	**0**	**28**	**56**	**0**	**28**	**56**
Coarse grits	7.7	7.3	7.0	7.5	7.5	6.2	7.3	7.7	5.8	7.2	4.5	4.7
Fine grist	7.7	...	6.5	7.7	...	6.5	7.2	...	7.1	7.3	...	4.5
Degerminated corn meal	7.5	7.7	6.2	6.8	7.6	6.7	7.2	7.7	6.2	6.3	4.7	4.3
Soy-fortified corn meal	7.0	...	6.3	7.0	...	6.3	6.7	...	6.7	7.0	...	6.7

[a]Stored at 49°C.

and fine grits and corn meal enriched with stabilized ferrous sulfate had reduced flavor scores, listed in the "objectionable" range. Soy-fortified corn meal with stabilized ferrous sulfate was judged "acceptable" after 56 days of storage.

The standard deviation for flavor scores in Tables V and VI is 0.47, and the LSD at 0.05 level would be 1.40.

Coarse and fine grits enriched with 100 mg iron/lb added as reduced iron (H) in starch and ferric orthophosphate had flavor scores above 7.0 after storage for 56 days at 49°C.

CONCLUSIONS

Apparently, such iron sources as reduced iron (E), reduced iron (H), and reduced iron (H) in starch can be added to common dry-milled corn products at levels of 40 mg iron/lb without appreciable segregation of iron or adverse changes in odors or flavors after accelerated storage. Stabilized ferrous sulfate added to these products at the same level did not segregate, but some deterioration of flavor and odor developed during storage. Soy-fortified corn meal was enriched at the 40-mg level with any of the four iron sources without problems. Ferric orthophosphate would probably also be acceptable if added at the 40-mg level.

All reduced iron sources, as well as stabilized ferrous sulfate, showed little segregation when used to enrich dry-milled corn products at 200 mg/lb; however, there was some degradation in flavor. Flavor scores of coarse and fine grits and of corn meal enriched with stabilized ferrous sulfate were less than acceptable.

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