Virginia Journal of Science
Volume 42, Number 3
Fall 1991

XPS Analysis of Reduced Iron Magnetically Extracted from Iron Fortified Breakfast Cereals

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ABSTRACT

Reduced iron powder was magnetically extracted from commercially available iron fortified breakfast cereals. X-ray photoelectron spectroscopy (XPS) studies determined that the outermost 5 nm of the extracted iron contained primarily carbon and oxygen with a small amount of iron and nitrogen. Argon and oxygen plasmas were used to remove the outer layer of organic contamination from the iron and increase the percentage of iron in the surface. The iron binding energy indicated that the outermost layer of the extracted iron was oxidized. The size of the iron particles was approximated from scanning electron microscope (SEM) photomicrographs.

INTRODUCTION

Cereal products are commonly enriched with iron to decrease the prevalence of anemia in the population (Baynes and Bothwell, 1990 and Fritz et al., 1975) and to replace naturally occurring iron that is lost during processing. Milling removes up to seventy-five percent of the iron phytate naturally occuring in wheat (Schroeder, 1971 and Ranum and Loewe, 1978). Iron enrichment of bread is mandatory in thirty-six states and in one territory, but fortification of breakfast cereal is voluntary (Reference Source, 1989).

Bioavailability is a measure of the ability of iron to be ingested by the body and varies with the chemical form of the iron, iron particle size, interaction of the iron with other diet components, chelation, and the body's need for iron (Ranum and Loewe, 1978). Standards for iron fortification are based upon iron quantity without accounting for differences in nutritional quality or bioavailability between iron sources (Davidson and Russo, 1976). Choice of a chemical form of iron for enrichment of a breakfast cereal requires a compromise between bioavailability, compatibility with the finished product, processing limitations and cost (Harrison et al., 1976). For example, ferrous sulfate has very high bioavailability but it may discolor or catalyze rancidity in breakfast cereals (Ranum and Loewe, 1978 and Davidson and Russo, 1976).

The most common source of iron used to enrich breakfast cereals is reduced iron powder (Ranum and Loewe, 1978 and Davidson and Russo, 1976). Reduced iron powder is dark metallic gray, insoluble in water, magnetic (Ranum and Loewe, 1978 and Davidson and Russo, 1976) and "generally recognized as safe" by the Food and Drug Administration for use as a dietary supplement (Code of Fed. Reg. part

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182.5375, 1990). Reduced iron powders of very small particle size are manufactured commercially by four different methods: high temperature reduction of ground iron oxides by hydrogen or carbon monoxide, electrolytic reduction of ferric iron to "electrolytic iron", and reaction of iron and carbon monoxide to form iron pentacarbonyl which is decomposed to give "carbonyl iron" and carbon monoxide (Ranum and Loewe, 1978). The ability of reduced iron particles to dissolve in the digestive system and be absorbed by the body is effected by the method of manufacture, particle size, solubility in dilute HCl, chemical impurities, surface area, porosity, and age (Ranum and Loewe, 1978). Oxidation of the iron surface or "rusting" decreases the availability of the iron to the body (Fritz et al., 1975 and Ranum and Loewe, 1978). Table 1 compares the relative bioavailability of several chemical forms of iron (Fritz et al., 1975).

Teflon®* -coated magnetic stir bars are used in the literature to recover reduced iron, added by researchers, from food suspended in water (Kadan and Ziegler, 1987 and Lee and Clydesdale, 1979) and from cereal in milk (Clydesdale and Nadeau, 1984). Kadan and Zeigler added reduced iron particles to powdered cereal mixes, processed the dry mix into breakfast flakes, recovered the iron magnetically, and used x-ray photoelectron spectroscopy (XPS) to determine the iron binding energy and to conclude that the iron surface was oxidized (Kadan and Ziegler, 1987). XPS provides the elemental composition of approximately the outermost 5 nm of a sample surface. X-rays of a characteristic energy irradiate the sample causing photoelectrons to be ejected and detected. The binding energy of an ejected electron is characteristic of the element to which the electron was bound. Small changes in the binding energy of an element, known as "chemical shifts", are due to differences in chemical bonding between atoms (Filbey and Wightman, 1991).

In this study, the reduced iron was extracted from breakfast cereals that are commercially available, rather than processed in the laboratory. The elemental composition of the outermost 5 nm of the iron particle surface and the chemical bonding between the surface atoms was characterized using XPS. Oxygen and argon plasma treatments were used to remove a layer of carbon and oxygen, determined to be carbohydrate cereal residue, from the iron surface. The size and shape of the iron particles was examined using the scanning electron microscope (SEM).

EXPERIMENTAL

The breakfast cereals in this study were Whole Wheat Total®**, Total Cornflakes, and Kellogg's Cornflakes purchased from a local grocery store. Total Cornflakes and Whole Wheat Total both contain 100% of the U.S. Recommended Daily Allowance (RDA) for iron per 28.4 grams (or 1 serving) and Kellogg's Cornflakes contains 10%***. The RDA of iron is 18 milligrams for adults and children four or more years of age (Code of Fed. Regs. part 104.47, 1990).

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* Teflon is a registered trademark of E. I. du Pont de Nemours & Co., Inc.

** Total is a registered trademark of General Mills, Inc.

*** Percentages obtained from nutrition information on breakfast cereal box.

TABLE 1. Bioavailability of Iron from Selected Sources (Fritz et al., 1975).

Iron Form	Particle Size	Relative Biological Value
Ferrous sulfate	---	100
Reduced iron		
H_2 reduction	10 - 20 microns	54
	325 mesh	34
	100 mesh	18
CO reduction	7 - 10 microns	36
	14 - 19 microns	21
	27 - 40 microns	13
Carbonyl iron	4 microns	69
	3 - 5 microns	69
	4 - 8 microns	64
Electrolytic iron	0 - 10 microns	76
	10 - 20 microns	75
	20 - 40 microns	48
	>40 microns	45
Ferric oxide (Rust)	---	4

Magnetic Extraction*

The breakfast cereal was crushed manually. Two clean 400 mL beakers and a clean 3.8 cm long Teflon-coated magnetic stir bar were rinsed with distilled, deionized H_2O (DD H_2O). One beaker was filled with DD H_2O and set aside. The magnetic stir bar was placed in the second beaker and enough crushed cereal was added to fill the beaker approximately two-thirds full. DD H_2O was added to fill the beaker. The cereal was allowed to absorb water and DD H_2O was added to refill the beaker. The cereal slurry was stirred magnetically for 20 to 30 minutes. The magnetic stir bar was carefully removed from the cereal slurry with non-metallic forceps and placed in the reserved beaker full of DD H_2O. The stir bar holding the extracted iron was magnetically stirred in the DD H_2O for several minutes to remove all visible cereal residue from the iron. The stir bar was removed with non-metallic forceps, placed on a clean watch glass, and dried in a 100°C oven for 10 to 15 minutes. A beaker of DD H_2O was magnetically stirred for 30 minutes as a control.

Once the iron was extracted from the cereal an effort was made to minimize exposure time of the iron to the atmosphere by proceeding immediately from extraction to plasma treatment to XPS analysis with minimal time delay. To

* Secondary school teachers should note that this experimental protocol lends itself to classroom demonstrations and science fair projects.

investigate the effect of the atmosphere, a sample of iron extracted from Total cornflakes was exposed to ambient conditions for two weeks prior to XPS analysis.

Plasma Treatment

The extracted iron was transferred from the magnetic stir bar to a small piece of ferro-type plate for plasma treatment. A Tegal Plasmod® was used to produce a radiofrequency (13.56 MHz)-generated 50 W plasma of argon or oxygen. The iron particles were treated in an argon plasma for various periods of time between 5 and 40 minutes or with an oxygen plasma for 5 minutes. The plasma treated iron powder was immediately secured on an XPS sample mount with double-sided transparent tape.

X-Ray Photoelectron Spectroscopy (XPS)

XPS analysis was performed with a Perkin-Elmer PHI 5300 spectrometer employing a Mg Kα (1253.6 eV) achromatic x-ray source operated at 15 keV with a power of 400 watts and a take-off angle of 90°. Survey scans were taken in the range of 0-1000 eV and narrow scans were obtained on any significant peaks observed in the survey scan spectra. The binding energy of each photopeak was referenced to C1s at 285.0 eV. The Perkin-Elmer 7500 computer (PHI software version 2.0) was used to obtain peak areas and for curve-fitting. The C1s region was curve-fitted with a full width at half maximum (FWHM) of 1.7± 0.1 eV and the O1s with FWHM of 2.0± 0.1 eV.

The iron powder magnetically extracted from the breakfast cereal was analyzed with XPS as removed from the cereal, after argon and oxygen plasma treatments, and after exposure to ambient conditions for 2 weeks. Manually crushed Whole Wheat Total cereal was also examined with XPS.

Scanning Electron Microscopy (SEM)

The extracted iron powder was secured on an SEM mount with double-sided transparent tape and sputter-coated with gold using an Edwards Sputter Coater S150B. An ISI-SX-40 SEM was used to obtain photomicrographs at 250X magnification. The iron particle size was approximated from three photomicrographs for each cereal.

RESULTS AND DISCUSSION

The magnetic iron particles extracted from the cereal clung together on the ends of the stir bar in strands. The very fine dark gray powder was insoluble in H_2O and dissolved in 3N HCl over a twenty-four hour period. Visual inspection showed no iron powder present on the control stir bar, a very small amount of iron for Kellogg's Cornflakes and a much greater amount for the two Total cereals. Approximately equal amounts of iron were removed from Whole Wheat Total and Total Cornflakes. These observations were anticipated as Total Cornflakes and Whole Wheat Total are fortified with 18 mg of iron per 28.4 grams (or 1 serving) and Kellogg's Cornflakes is fortified with only 1.8 mg of iron per 28.4 grams.

The XPS calculated atomic percentages are presented in Table 2 for each element in the surface of the magnetically extracted iron powder. These results show that the outermost 5 nm of the iron as extracted from the breakfast cereal contains primarily carbon and oxygen and less than 5% iron and nitrogen. The

TABLE 2. Elemental Composition of the Outermost 5 nm of the Reduced Iron Magnetically Extracted from Breakfast Cereal.

Carrier Cereal	Plasma Treatment	Atomic Concentration (%) C1s	O1s	Fe2p	N1s
Whole Wheat Total	No plasma*	70	25	3	2
	Ar - 5 min.	83	17	0	---**
	Ar - 40 min.	63	28	6	3
	O_2 - 5 min.	34	50	16	0
Total Cornflakes	No plasma	78	20	1	1
	No plasma (2 wks. air exposure)	60	35	4	1
	Ar - 20 min.	84	13	1	2
	Ar - 40 min.	66	26	5	3
	O_2 - 5 min.	48	39	12	1
Kellogg's Cornflakes	No plasma	73	23	2	2
	Ar - 30 min.	81	15	1	3
	O_2 - 5 min.	51	38	10	1

* Iron as extracted from the breakfast cereal
** N1s region not scanned

intensities of the Fe2p photopeaks of the iron powdesr, as extracted from the breakfast cereals, were low due to the small percentage of iron in the surfaces. The iron valence state could not be positively determined. Plasma treatment was used to remove an outer layer of organic contamination and expose more iron for characterization.

Argon plasma, even up to 40 minutes, followed by exposure to the atmosphere was relatively ineffective in cleaning the iron surface. There is evidence in the literature that an argon plasma generates stable free radicals in the sample surface which react with room air upon exposure. Carbon and oxygen redeposit on the surface because there is no mechanism for converting the molecular fragments removed from the surface into permanently volatile compounds (Liston, 1989). A purge of hydrogen following plasma treatment might greatly increase the cleaning effectiveness of the argon plasma, but was not attempted. Oxygen plasma treatment for only 5 minutes removed the organic contamination and resulted in a 500% or greater increase in the concentration of surface iron as given in Table 2. An oxygen plasma is capable of oxidizing the carbon and oxygen fragments ablated from the sample surface into volatile species such as CO, CO_2 and H_2O (Liston, 1989). The effect of the argon and oxygen plasma treatments on the Fe2p photopeaks of the reduced iron magnetically extracted from Whole Wheat Total, Total Cornflakes, and Kellogg's Cornflakes are shown in Figures 1, 2, and 3, respectively.

The C1s and O1s regions are shown in Figures 4, 5 and 6 and the respective curve-fitting results are tabulated in Tables 3 and 4. The most intense C1s photopeak was positioned at 285.0 eV and is assigned to CHx. Three higher binding

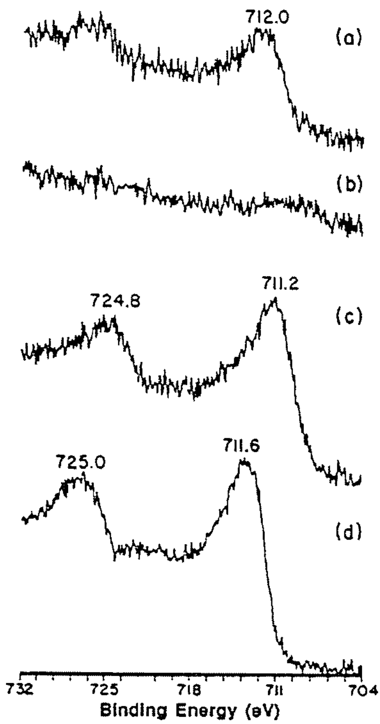

FIGURE 1. XPS Fe2p Region of Reduced Iron Magnetically Extracted from Whole Wheat Total Breakfast Cereal. (a) No plasma treatment. (b) 5 min. Ar plasma treatment. (c) 40 min. Ar plasma treatment. (d) 5 min. O2 plasma treatment.

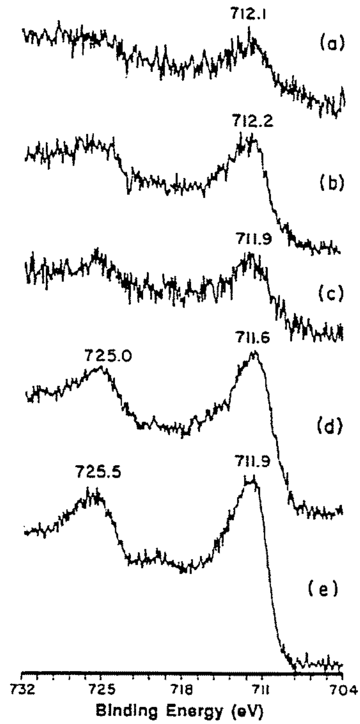

FIGURE 2. XPS Fe2p Region of Reduced Iron Magnetically Extracted from Total Cornflakes Breakfast Cereal. (a) No plasma treatment. (b) 2 weeks air exposure and no plasma treatment. (c) 20 min. Ar plasma treatment. (d) 40 min. Ar plasma treatment. (e) 5 min. O2 plasma treatment.

energy photopeaks were present and represent different carbon and oxygen environments. The photopeaks at approximately 286.6 eV, 287.9 eV and 289.0 eV are assigned to C-O, O-C-O and/or C=O, and O-C=O bonding, respectively (Clark et al., 1978). When the iron extracted from Whole Wheat Total was treated in an argon plasma for 5 minutes, a peak appeared at 283.7 eV and could not be positively identified. The O1s region contained a C-O photopeak at approximately 533.7 eV, a C=O photopeak around 532.8 eV and an iron oxide photopeak at approximately530.7 eV (Clark et al., 1978). The intensity of the O1s iron oxide peaks increased as the intensity of the Fe2p photopeaks increased. The C1s and O1s regions of the crushed Whole Wheat Total cereal shown in Figure 7 correlate well with the carbon and oxygen on the iron surface shown in Fig. 4a and suggest that the iron powder extracted from the whole wheat Total was coated with a layer of carbohydrate cereal residue.

The iron extracted from Total Cornflakes and exposed to room air for two weeks became faintly dark orange in color and XPS analysis showed an increase in the amount of iron in the surface relative to the amounts of carbon and oxygen. The percentage of iron in the surface increased as the outer layers "rusted" or corroded

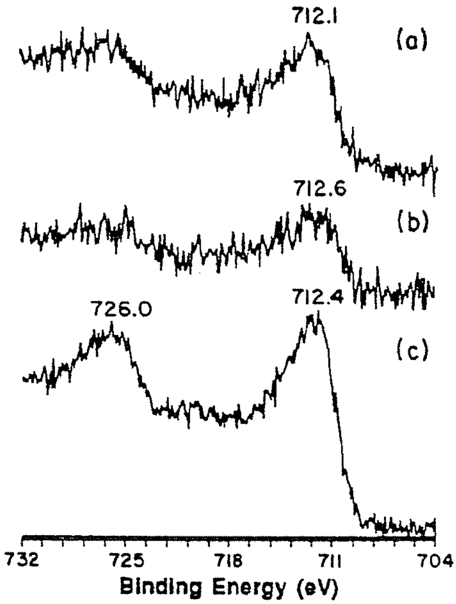

FIGURE 3. XPS Fe2p Region of Reduced Iron Magnetically Extracted from Kellogg's Cornflakes Breakfast Cereal. (a) No plasma treatment. (b) 30 min. Ar plasma treatment. (c) 5 min. O_2 plasma treatment.

TABLE 3. C1s Curve-fitting Results for Reduced Iron Magnetically Extraced from Breakfast Cereals.

Carrier Cereal	Fig.	Plasma Treatment	Atomic Concentration (%) Binding Energy (eV) 289.0	287.9	286.6	285.0	283.7
Whole Wheat Total	4a	No plasma*	6	5	20	69	--
	4b	Ar - 5 min.	5	3	12	65	15
	4c	Ar - 40 min.	7	4	17	72	--
	4d	O_2 - 5 min.	11	6	20	63	--
Total Cornflakes	5a	No plasma	5	3	19	73	--
	5b	No plasma (2 wks air exposure)	4	7	33	55	--
	5c	Ar - 20 min.	8	--	18	74	--
	5d	Ar - 40 min.	7	1	14	77	--
	5e	O_2 - 5 min.	9	2	20	50	--
Kellogg's Cornflakes	6a	No plasma	9	2	20	69	--
	6b	Ar - 30 min.	8	--	18	75	--
	6c	O_2 - 5 min.	7	4	21	69	--

* Iron extracted from the breakfast cereal

TABLE 4. O1s Curve-fitting Results for Iron Magnetically Extracted from Breakfast Cereals.

Carrier Cereal	Fig.	Plasma Treatment	Atomic Concentration (%) Binding Energy (eV) 535.5	533.7	532.8	530.7	528.7
Whole Wheat Total	4a	No plasma*	2	42	36	19	--
	4b	Ar - 5 min.	--	31	46	20	4
	4c	Ar - 40 min.	--	27	45	28	--
	4d	O_2 - 5 min.	--	16	40	44	--
Total Cornflakes	5a	No plasma	3	49	36	12	--
	5b	No plasma (2 wks. air exposure)	--	57	22	21	--
	5c	Ar - 20 min.	--	42	50	8	--
	5d	Ar - 40 min.	--	19	51	30	--
	5e	O_2 - 5 min.	--	17	32	51	--
Kellogg's Cornflakes	6a	No plasma	--	27	57	15	--
	6b	Ar - 30 min.	--	45	48	7	--
	6c	O_2 - 5 min.	--	15	40	44	--

* Iron as extracted from the breakfast cereal

TABLE 5. Fe2p Region Characteristic Binding Energies.

Sample Description		Fe2p3/2 Binding Energy (eV)	Fe2p1/2 and Fe2p3/2 Separation (eV)
Fe^{o}		710.0[a]	13.2[b]
Fe_2O_3		712.6[a]	13.6[b]
Reduced iron magnetically extracted from:			
Whole Wheat Total	No plasma treatment	712.0	---
	Ar plasma - 5 min.	---	---
	Ar plasma - 40 min.	711.2	13.6
	O_2 plasma - 5 min.	711.6	13.4
Total Cornflakes	No plasma treatment	712.1	---
	No plasma treatment (2 wks. air exp)	712.2	---
	Ar plasma - 20 min.	711.9	---
	Ar plasma - 40 min.	711.6	13.4
	O_2 plasma - 5 min.	711.9	13.6
Kellogg's Cornflakes	No plasma treatment	712.1	---
	Ar plasma - 30 min.	712.6	---
	O_2 plasma - 5 min.	712.4	13.6

a. Kadan and Ziegler, 1987
b. Waghorn, 1979

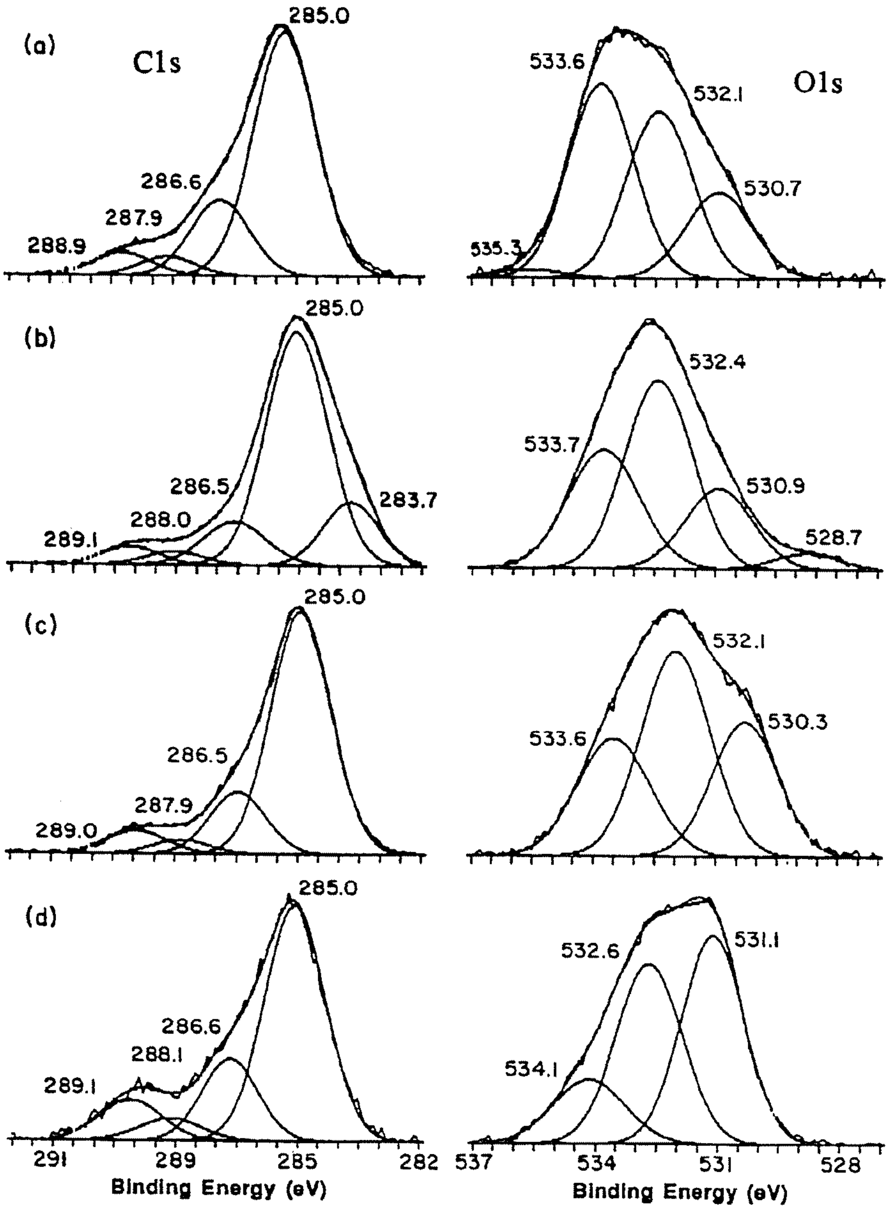

FIGURE 4. Curve-fit C1s and O1s Regions of Reduced Iron Magnetically Extracted from Whole Wheat Total Breakfast Cereal. (a) No plasma treatment. (b) 5 min. Ar plasma treatment. (c) 40 min. Ar plasma treatment. (d) 5 min. O2 plasma treatment.

increasing the total surface area of the particle by "pitting" and exposing iron that lay just below the surface. Table 5 compares the binding energies of reduced iron and ferric oxide found in the literature (Kadan and Ziegler, 1987 and Waghorn, 1979) to all well resolved Fe2p photopeaks. Both the binding energies of the

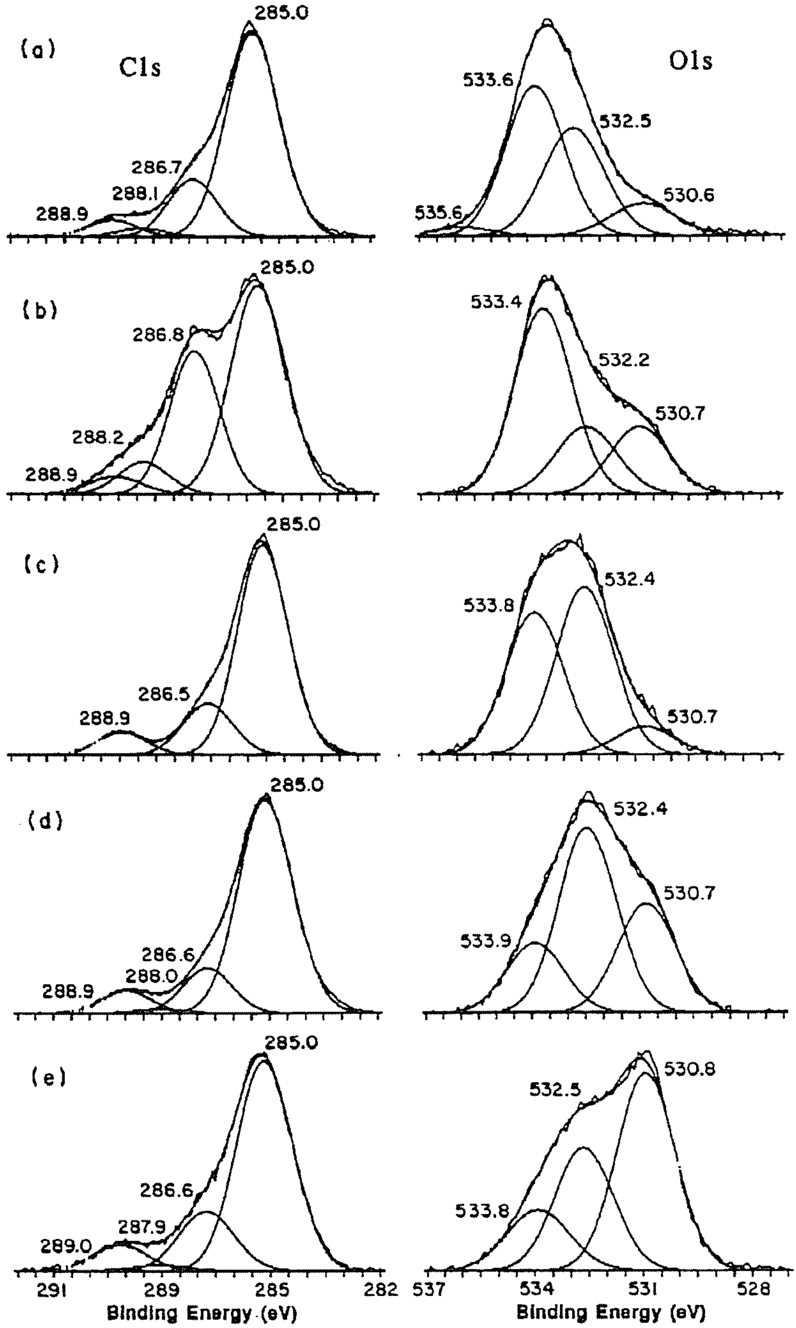

FIGURE 5. Curve-fit C1s and O1s Regions of Reduced Iron Magnetically Extracted from Total Cornflakes Breakfast Cereal. (a) No plasma treatment. (b) 2 weeks air exposure and no plasma treatment. (c) 20 min. Ar plasma treatment. (d) 40 min. Ar plasma treatment. (e) 5 min. O2 plasma treatment.

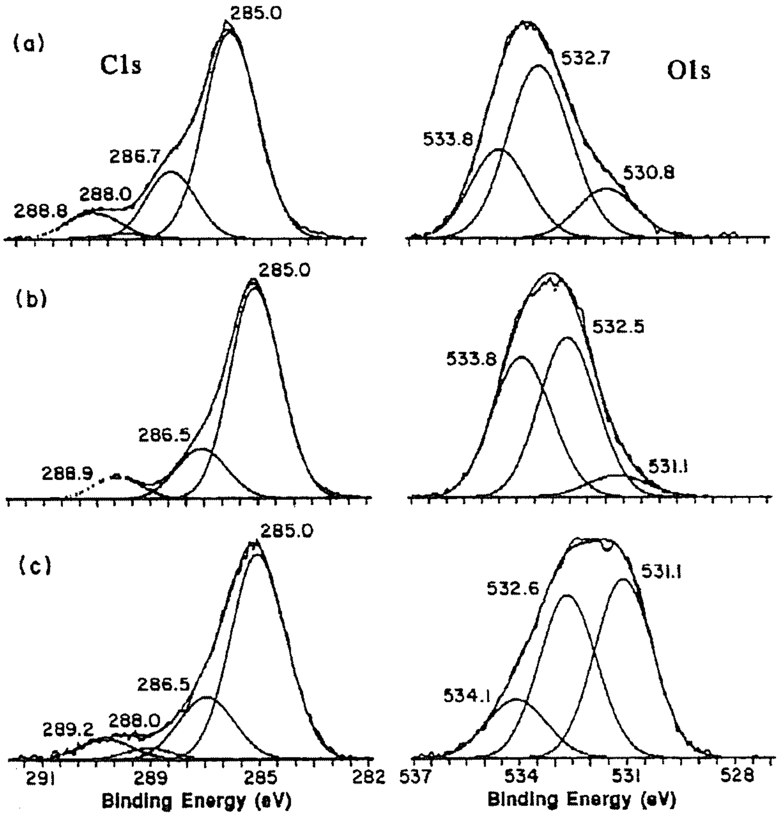

FIGURE 6. Curve-fit C1s and O1s Regions of Reduced Iron Magnetically Extracted from Kellogg's Cornflakes Breakfast Cereal. (a) No plasma treatment. (b) 30 min. Ar plasma treatment. (c) 5 min O2 plasma treatment.

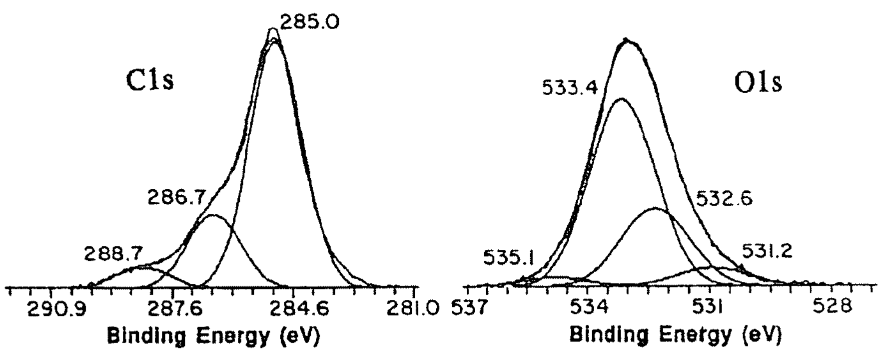

FIGURE 7. Curve-fit C1s and O1s Regions of Crushed Whole Wheat Total Breakfast Cereal.

Fe2p3/2 photopeaks and the separation between the Fe2p3/2 and Fe2pl/2 photopeaks of the iron powder as extracted from the cereal, after exposure to the atmosphere for two weeks, and after argon or oxygen plasma treatment are closer to the literature values for ferric oxide than for reduced iron. Therefore, the XPS analysis showed that the outer molecular layers of the reduced iron, as extracted from the breakfast cereal, were oxidized. However, because the Fe2p binding energies were slightly less than the literature values for ferric oxide (Fe_2O_3), iron oxides in both the Fe^{2+} and the Fe^{3+} valence states might be present on the iron surface. The Fe2p3/2 binding energy of ferrous oxide (FeO), in the Fe^{2+} valence state, would be shifted above that of reduced iron, but would not be as high as the Fe2p3/2 binding energy of ferric oxide, in the Fe^{3+} valance state (Waghorn, 1979). The presence of a combination of iron oxides, in both the Fe^{2+} and Fe^{3+} valence states, would account for the Fe2p3/2 binding energies of the extracted iron particles listed in Table 5, but was not confirmed.

There is no way to conclude from this study whether or not the iron surface was oxidized while in the cereal or if it became oxidized during the magnetic extraction and drying procedure. It has been shown in the literature that the surface of reduced iron is oxidized even before it is added to foods (Kadan and Ziegler, 1987). The extreme oxidizing effect of the oxygen plasma treatment did not change the binding energy of the Fe2p photopeaks which supports the proposal that the iron was oxidized before it was plasma treated. Reduced iron and ferric ferrous oxide (Fe_3O_4) are magnetic, but Fe_2O_3 and FeO are not magnetic. All extracted iron samples remained magnetic after extraction, plasma treatment, and XPS analysis. Therefore, only the outermost molecular layers of the extracted iron powder contained Fe_2O_3 and FeO, but the interior was magnetic iron.

SEM photomicrographs of the extracted iron magnified 250X revealed that the particles were rough and irregular in shape, resembling pieces of gravel. The iron particles ranged from approximately 8 to 80 microns in diameter.

SUMMARY

The relative amount of reduced iron powder magnetically extracted from three iron fortified breakfast cereals, Whole Wheat Total, Total Cornflakes and Kellogg's Cornflakes, corresponded to the level of iron enrichment as calculated from the % U.S. RDA supplied by the cerial. The extracted iron particles were coated with carbohydrate cereal residue which was most effectively removed with a 5 min. oxygen plasma treatment. XPS studies showed that the surface of the reduced iron powder as extracted from the breakfast cereal was oxidized. Plasma treatment and exposure of the iron particles to the atmosphere increased the percentage of iron in the surface, but did not effect the binding energy of the Fe2p photopeaks corresponding to oxidized iron.

ACKNOWLEDGEMENT

The help of Frank Cromer with the XPS and SEM work is appreciated. Ms. Heisey performed all experimental work and prepared this manuscript. Dr. Burch contributed the magnetic extraction procedure. Dr. Wightman provided technical assistance with the experiments and with this paper.

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