Detection and Measurement of Iron Compounds in Fortified Flours

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Wheat Flour Fortification Legislation

September 2013: 77 countries require iron and/or folic acid in wheat flour

All countries fortify flour with at least iron and folic acid except Australia which does not include iron, and Nigeria, Venezuela, the United Kingdom, and the Philippines which do not include folic acid.
## 2008 Workshop Recommendations

<table>
<thead>
<tr>
<th>Nutrient</th>
<th>Type of flour (extraction)</th>
<th>Fortificant</th>
<th>Level of nutrient to be added (parts per million)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>By per capita wheat flour intake (g/day)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>&lt;75 g/day</td>
</tr>
<tr>
<td>Iron</td>
<td>Low</td>
<td>NaFeEDTA</td>
<td>40</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sulfate/Fumarate Electrolytic</td>
<td>60</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>NR</td>
</tr>
<tr>
<td></td>
<td>High</td>
<td>NaFeEDTA</td>
<td>40</td>
</tr>
<tr>
<td>Zinc</td>
<td>Low</td>
<td>Zinc Oxide</td>
<td>95</td>
</tr>
<tr>
<td></td>
<td>High</td>
<td>Zinc Oxide</td>
<td>100</td>
</tr>
<tr>
<td>Folic Acid</td>
<td>Low or High</td>
<td>Folic Acid</td>
<td>5.0</td>
</tr>
<tr>
<td>Vitamin B12</td>
<td>Low or High</td>
<td>Cyancobalamin</td>
<td>0.04</td>
</tr>
<tr>
<td>Vitamin A</td>
<td>Low or High</td>
<td>Vitamin A palmitate</td>
<td>5.9</td>
</tr>
</tbody>
</table>
Recommendations on Wheat and Maize Flour Fortification
Meeting Report: Interim Consensus Statement

Available in UN languages

English
Russian
Chinese

Suggested citation


(http://www.who.int/nutrition/publications/micronutrients/wheat_maize_fo rt.pdf, accessed [date]).
## Success of Fortifying with Iron

<table>
<thead>
<tr>
<th>Country</th>
<th>Population studied</th>
<th>Improvement?</th>
</tr>
</thead>
<tbody>
<tr>
<td>China</td>
<td>Women</td>
<td>Yes</td>
</tr>
<tr>
<td>Iran</td>
<td>Women and men</td>
<td>Yes</td>
</tr>
<tr>
<td>Venezuela</td>
<td>School-age children</td>
<td>Yes</td>
</tr>
<tr>
<td>Fiji</td>
<td>Women of child-bearing age</td>
<td>Yes</td>
</tr>
<tr>
<td>Azerbaijan</td>
<td>Preschool and school-age children</td>
<td>Yes</td>
</tr>
<tr>
<td>Kazakhstan</td>
<td>Preschool and school-age children</td>
<td>Yes</td>
</tr>
<tr>
<td>Mongolia</td>
<td>Preschool and school-age children</td>
<td>Yes</td>
</tr>
<tr>
<td>Tajikistan</td>
<td>Preschool and school-age children</td>
<td>Yes</td>
</tr>
<tr>
<td>South Africa</td>
<td>Women of child-bearing age</td>
<td>No</td>
</tr>
<tr>
<td>Uzbekistan</td>
<td>Preschool and school-age children</td>
<td>No</td>
</tr>
</tbody>
</table>

FFI review May 2013. See references on last slides.
Vitamin and Mineral Deficiency Contributes to:

• More than one-third of all deaths in children under the age of 5

• Stunting of an estimated 195 million children under age 5 in developing countries

• Undeveloped cognitive capacity, productivity and earning potential
Iron Deficiency:

- Affects *more people* than any other health condition
- Reduces *work capacity*
- Impairs a child’s physical and *intellectual development*
- Contributes to 20% of all *maternal deaths*
- Is a leading cause of anemia which affects *2 billion people* – over 30% of the world’s population


Photo by Ivan Mateev at iStockphoto
Micronutrients that can be added to flour

- **Vitamins**
  - A,
  - B group (B1, B2, B3, B6, B12)
  - Folic Acid
  - D

- **Minerals**
  - Iron, Calcium, Selenium, Zinc
Micronutrients for flour

• Minerals
  – Iron; Electrolytic, Ferrous Fumarate, Ferrous Sulphate, NaFeEDTA
  – Calcium; Calcium Carbonate or Sulphate
  – Magnesium; Magnesium Sulphate or Oxide
  – Phosphorus; Calcium Phosphate
  – Zinc; Zinc Sulphate or Oxide
Electrolytic Iron Specification

• Must USP/FCC grade, very fine particle size

• Assay
  – 96.0% Fe minimum
  – Particle Size Thru 200 mesh 99% min, Thru 325 mesh 95%
  – Arsenic 8ppm, Lead 25ppm, Mercury 5 ppm maximum for all above
Ferrous Sulphate

• Dried, Tan powder meeting USP/FCC grade
• Assay
  – As FeSO₄ 86-89% As Fe 31.6-32.6%
  – Particle size Thru 100 mesh 99.5%, Thru 200 mesh 90%
  – Arsenic 3 ppm, Lead 10 ppm, Mercury 3 ppm maximum for all above
Ferrous Fumarate

• Dried, Dark Tan powder meeting USP/FCC grade

• Assay
  – As FeFumarate 95-98% As Fe 31.6-32.6%
  – Particle size Thru 100 mesh 99.5%, Thru 200 mesh 90%
  – Arsenic 3 ppm, Lead 10 ppm, Mercury 3 ppm (maximum for all heavy metals above)
Sodium Iron EDTA

• Yellow Green Powder

• Assay
  – As EDTA 65.5-70.5%, As Iron 12.5-13.5%
  – Arsenic 1 ppm, Lead 1 ppm max
  – Particle Size Through 100 mesh 99.5%

• NOTE: Sodium Iron EDTA is recommended for high extraction wheat flour and any maize flour or maize meal
## Properties of Iron Compounds

<table>
<thead>
<tr>
<th>Iron source</th>
<th>Conc %Fe</th>
<th>Cost $/kg</th>
<th>Cost $kg Fe</th>
<th>Colour</th>
<th>Magnetic</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ferrous Sulphate</td>
<td>32</td>
<td>1.30</td>
<td>4.06</td>
<td>Tan</td>
<td>No</td>
</tr>
<tr>
<td>Ferric O. Phosph</td>
<td>29</td>
<td>2.50</td>
<td>7.81</td>
<td>Red</td>
<td>No</td>
</tr>
<tr>
<td>Iron, H Reduced</td>
<td>97</td>
<td>1.75</td>
<td>1.80</td>
<td>Black</td>
<td>Yes</td>
</tr>
<tr>
<td>Iron, E Reduced</td>
<td>98</td>
<td>4.00</td>
<td>4.10</td>
<td>Black</td>
<td>Yes</td>
</tr>
<tr>
<td>NaFe EDTA</td>
<td>13</td>
<td>6.00</td>
<td>46.15</td>
<td>Tan</td>
<td>No</td>
</tr>
</tbody>
</table>
Process Control in Flour Milling

• Flour milling needs process controls to ensure consistent quality and safety of the output: flour adequately milled to meet market requirements and safety standards.
• Effective process control systems use mechanisms to monitor activities and take timely corrective action.
• Well implemented process control gives an early warning of problems which in turn helps to avoid wastage, reworking of product, customer complaints, food recalls and liability issues etc.
• Good Process Control systems include multiple measurable parameters – they do not rely on just one parameter
QC/QA testing
Qualitative vs Quantitative

• QC/QA testing is used as just one of the tools available to the miller – used in conjunction with other process control tools
• Use of one parameter, flour additive or micronutrient as indicator for rapid qualitative/semi quantitative testing
Chemical Analysis of Micronutrients
Quantitative Methods

• Vitamins
  – HPLC, GC, Spectrophotometric, Colorimetric methods

• Minerals
  – Atomic Absorption Spectroscopy, Colorimetric and Spectrophotometric Methods

NOTE: QUANTITATIVE METHODS MEASURE TOTAL LEVELS OF VITAMINS AND MINERALS i.e NATURAL AND ADDED LEVELS
Chemical Analysis of Micronutrients
Qualitative Methods

• Vitamin A
  – Colorimetric method for maize flour developed in South Africa

• Iron
  – Spot Test Method for added Iron in maize and wheat flours

NOTE: Both methods measure added micronutrients but not Intrinsic iron in flour before fortification
Relative Costs of Chemical Assay (testing costs only)

- **Vitamins Analysis**
  - Expensive: $25-50 per vitamin per sample and on different equipment
  - Assume 20 mills, one sample per week for 5 micronutrients at average price of $30 per sample – Total cost: $156,000 per year

- **Mineral Analysis**
  - Less Expensive: $15 per sample for Iron
  - Assume 20 mills one sample per week @ $15 per sample – Total cost: $15,600
Use of Representative Micronutrient for Chemical Assay

- In flour fortification premixes are used containing vitamins and minerals (Iron)
- Iron is cheaper and more accurate to measure in flour than many vitamins
- Iron can be the representative micronutrient of the premix added to flour for chemical assay
- Iron can also be used in Spot Test for monitoring in the country AND as QC tool at the mill
- This system requires premix supplier to provide Certificate of Analysis (CoA) and importer to audit CoA
Iron Spot Test: Reagents

- Potassium Thiocyanate KSCN
- Hydrochloric Acid
- Hydrogen Peroxide
Iron Spot Test: Modification for Sodium Iron EDTA

• Add the 50/50 solution of Potassium Thiocyanate (KSCN) 10% solution W/V and Hydrochloric Acid 2N solution V/V onto the flour slick

• Allow the spots to develop over 2 minutes and compare to standard.

• Omit the use Hydrogen Peroxide solution
Iron Spot Test: Additional points

• When comparing samples to standards the same type of iron compound MUST used in both the sample and the standard i.e. FeSO4 for FeSO4 or Electrolytic Fe for Electrolytic Fe and NaFeEDTA standard for NaFeEDTA

• Do not use FeSO4 standard to evaluate flour fortified with Electrolytic Fe

• Differences due to compound type and particle size.
Mill QC: Iron Spot Test

Iron Spot Test for added premix in flour
Mill QC: Iron Spot Test
Recent Developments

• Millers Fortification Toolkit Updated
  – Additional information and video clips based on new technologies, feeders, QC tests
  – Was be posted on line in March 2013

• Iron Spot Test modified for NaFeEDTA
  – Official AACC method did not detect NaFeEDTA
  – Test modified to detect NaFeEDTA – simple modification

• iCheck hand held device can measure Iron as NaFeEDTA quantitatively
Thank you for your kind attention!

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