International Journal of Chemistry and Pharmaceutical Sciences

Journal Home Page: www.pharmaresearchlibrary.com/ijcps

Research Article **Open Access**

Qualitative and quantitative measurement of iron in wheat flour of some flour companies of Golestan province by Spectroscopic method

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A B S T R A C T

Iron is one of the most abundant elements in nature but iron deficiency due to low absorption in the human digestive system is one of the most common nutritional problems. Therefore, consumption of food fortified with these nutrient deficiency problems can be compensated for it. Bread as the main Persian food is the perfect tool to achieve this goal. Uv-Vis absorption spectroscopy method is one of the most reliable methods to precisely measure of iron in cereals. in this project, after collecting samples of flour from the regions, the existing iron released by digestion and Complex Fe-SCN was formed in the presence of thiocyanat ions. Then quantitatively measured by absorption spectroscopy method based on the calibration curve. Also a simple and inexpensive method in which show the presence of iron is indicating for qualitative measurement before each quantitative test was performed on the samples. LOD, LOQ and RSE this method were 0/0008, 0/0029 and 0/664 (mg / L), respectively, in the 0/00175 – 0/00075 range concentrations.

Keywords: Iron determination, Thiocyanate, Absorption spectroscopy, Fortified food products, Flour

A R T I C L E I N F O

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Article History: Received 11 October 2016, **Accepted** 24 November 2016, **Available Online** 27 December 2016

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Manuscript ID: IJCPS3277

PAPER-QR CODE

Citation: Elham baher. Qualitative and quantitative measurement of iron in wheat flour of some flour companies of Golestan province by Spectroscopic method. *Int. J. Chem, Pharm, Sci.*, 2016, 4(12): 660-663.

1. Introduction

Now Bread has a significant role in feeding of worldwide people. Intake of some micronutrients such as zinc, thiamine, iron, and calcium from food is remarkable, however, due to the high degree of extraction of the fiber, especially in the Persian bread, the availability of these elements is questioned especially iron and zinc [1-7].

The degree of flour extraction, fermentation and baking (baking temperature and time) is effective on the availability of elements, especially iron, calcium and zinc, therefore, to enhance the nutritional value and improve the quality of bread snacks, traditional fermentation is recommended with the appropriate procedures and in the process of making bread [2, 4, 5].

Iron deficiency is the most common cause of anemia, and according to global statistics nearly a quarter of the world's population are anemic and it can be seen in all ages [3, 4]. Women in childbearing age and children under 5 years are more susceptible to anemia and iron deficiency in these age groups could lead to delayed physical growth, reduced IQ and learning as much as 5 to 10 points on them. People with iron deficiency and anemia become sicker and longer course of their disease, due to the weakening of the immune system and reduces the body's resistance to infection. One of the ways to preventing iron deficiency is the fortification of foods.

Wheat flour fortifications in the United States and Britain have been implemented 60 years ago and micronutrients such as iron, calcium and B vitamins added to flour. Since 1996, many countries are also added folic acid to wheat flour [5, 6]. The reason for this, role of folic acid in reducing congenital neural tube defects (NTD), a decrease in the prevalence of high homocysteine (a risk factor for cardiovascular disease), reduced incidence of cancer and Alzheimer's disease that has proven in several studies [5, 8].

The most important principle for quality control of enriched product in order to ensure the availability of added micronutrient is using methods that is simple possibility of doing it in the factory and neither expensive nor time-consuming [11-13]. One of these tests, that can be performed daily and regularly in flour factories, is qualitative method with which performed by using color intensity of samples after the addition of proper reagents. In order to determine the exact amount of iron in flour, a number of samples is transferred to the laboratory and measured by Uv-Vis absorption spectroscopy method [12-20].

Uv-Vis absorption spectroscopy is a simple, quick, inexpensive and sensitive for quantitative analysis of iron and other nutrients. In the present work, first the qualitative then quantitative method were performed on samples of flour, that obtained results shows the simplicity and accuracy of tests. Also, for the first time thiocyanate as an inexpensive ligand was used for complex making of iron in both qualitative and quantitative method.

2. Materials and Methods

Whatman Filter paper # 1, Manual sieve, Watch glass, Hydrochloric acid (HCl) 37%, Hydrogen peroxide (H_2O_2) 3% v/v, Potassium Thiocyanate-10%, Iron solution0.1 mgL^{-1}, Thiocyanate 0.01M, Hydroxylamine hydrochloride-10%, Acetate buffer(PH=4.5). All materials were purchased from the Fluka company (Germany).Three types of flour samples were collected from different sources (Fajr, Barekat and Kalaleh). For quantitative measurements, all absorption spectra were made by using UV/Vis spectrometer (PG Instruments Ltd, China) equipped with a 5.0 mm quartz cell. This spectrophotometer has a wavelength accuracy of ±0.2 nm with wavelength range of 200–900 nm.

Procedure

First, in order to qualitative analysis of iron, the filter paper was placed over the watch glass; the surface of the filter paper was thoroughly wet with potassium thiocyanate solution. The flour sample was sifted uniformly on the surface of filter paper by a hand sieve. Then acidic solution of potassium thiocyanate (thiocyanate 5% in HCl) was sprayed over the flour layer. In the next step, small amounts of the H_2O_2-solution was added to the flour and after a few minutes the reaction of oxidation of iron completed and red spots indicated that the presence of iron. **Figure**. 1 shows the qualitative analysis steps.

Figure 1: Qualitative analysis steps

To prepare standard solutions for quantitative analysis, solutions 1 to 7, were respectively maked, with an increase of 0, 5, 15, 20, 30, 40 and 50 ml of iron (0.01mg / L) in 100 ml volumetric flasks. 2 ml HCl was added to each flask then was adjusted to volume with deionized water. 10 ml from each of them was transferred to volumetric flasks (25 mL) numbers of 8 to 14 along with 1 ml hydroxylamine hydrochloride, 5 ml acetate buffer and 2 ml of thiocyanate were added to them then were adjusted to volume with deionized water. After solution preparation, their absorption was measured with a spectrophotometer T90 UV/Vis -PG at a wavelength 240 nm.

Sample preparation

First, 5 ml of hydrochloric acid was poured into the porcelain crucible containing the ashes of flour. After stirring, porcelain crucible was placed on water bath to evaporate acid**.** Again, 2 ml hydrochloric acid was added to porcelain crucible and another 5 minutes was placed on the water bath. Then material was filtered and transferred into volumetric flasks (100 mL) and adjusted to volume with

deionized water. 20 ml aliquot was pipet into 50 ml volumetric flask, and 2 ml hydroxylamine HCl was added to solution. After 5 min, 10 ml buffer solution and 2 ml Thiocyanate were added and dilute to volume, then absorbance of iron was measure by spectrophotometer at 240 nm.

3. Results and Discussion

Qualitative determinations of iron

Flour samples were uniformly placed on filter paper while it was spiked with a solution of potassium thiocyanate. Then the thiocyanate acid and hydrogen peroxide solution was added to the flour layer. After a few minutes the oxidation reaction is completed and red spots was appeared in the presence of iron in flour. The oxidation reaction was carried out as follows:

$$2\,[Fe(SCN)_6]^{4-} + H_2O_2 + 2\,H^+ \rightarrow 2\,[Fe(SCN)_6]^{3-} + 2\,H_2O$$

Experiment's pictures of the flour samples; Barekat, Fajr and Kalaleh were shown in **Figure** 2 (a), 2(b) and 2(c) states, respectively.

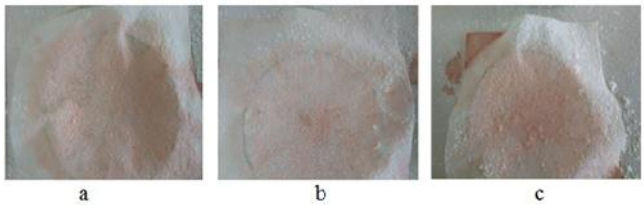

Figure **2:** Qualitative analysis's pictures of iron in flour samples Barekat (a), Fajr (b) and Kalaleh (c)

Quantitative determinations of iron

Solutions 8 to 14 were prepared as described and their absorption was read three times in the wavelength 240nm and was used to plot the calibration curve. **Figure** 3 shows the calibration curve plot that including absorbance value of standard solutions against of concentration with correlation coefficient 0/95.

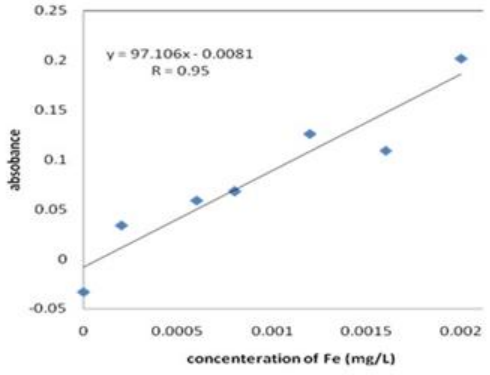

Figure **3:** Calibration curve in the range of concentration (0.00 – 0.002 mg / L)

The amount of iron in flour samples Barekat, Fajr and Kalale are 0.0037, 0.0008 and 0.0034 mg / L respectively. According to the results, Fajr flour and Barekat flour have the lowest and highest amounts of iron respectively.

The validation of the method

The sensitivity of method was assessed by limit of detection (LOD) and limit of quantification (LOQ) that were calculated through the calibration curve. The limit of detection (LOD) is the lowest amount of analyte in a sample that can be detected at a certain level of confidence. LOQ is the lowest amount of analyte in a sample that can be measured by the method. The limit of detection, defined as LOD= $3S_b/m$ (where S_b and m are standard deviation of the blank and slope of the calibration graph, respectively) was 0.0008 mgL^{-1}. The limit of quantification, defined as LOQ= 10 LOD was 0.0028 mgL^{-1}. The precision of the method, calculated as the relative standard deviation (RSD) of three independent measurements carried out 0.664 µg L^{-1}. The calibration graph was linear in the range of 0.0075–0.00175 $mgL^{-1}$with a correlation coefficient of 0.95.

Linear dynamic range (LDR) is the concentration range which the calibration curve is linear and the broader LDR is better. To determine the LDR of method, solutions with similar proportions are prepared from the most diluted and most concentrated calibration solution series. Then absorption of nine resulting solution were measured in 240 nm. Then calculated concentration is displayed according to curve equation in terms of the concentration on the Y axis and the actual amount concentration on the X axis. LDR is determined to draw the best line that passes from results. Calculated merit numbers are shown in Table 1.

Table 1: Merits number of the proposed method

LDR (mgL^{-1})	RSD (%)	LOQ (mgL^{-1})	LOD (mgL^{-1})	R
0.0007-0.0017	0.664	0.0028	0.0008	0.95

4. Conclusions

Spectrophotometry is an inexpensive, simple, and high-precision measurement of trace elements, especially iron in foods that play an important role in human health. Iron-thiocyanate complex, which formed by the proposed method, shows measured amount of iron in flour samples well. Also, the use of thiocyanate as a ligand reduces costs while has similar results with ligands such as phenanthroline in previous work. Also suggested qualitative methods are in good agreement with the obtained results by using spectrophotometry and easily are carried out in the factory.

5. Acknowledgment

This research was supported by Golestan University. We would also like to show our gratitude to Dr. Maryam Vaezi for assistance and comments that greatly improved the manuscript.

6. References

[1] Global Alliance for Improved Nutrition, Micronutrient Initiative, Flour Fortification Initiative. Recommendations on wheat and maize

flour fortification. Meeting report: interim consensus statement. Geneva: World Health Organization, FAO, UNICEF; 2009 [cited 2010 Feb 28].

[2] R.FHurrell. Preventing Iron Deficiency through Food Fortification: Nutrition reviews, 1997,55(6): 210-222.

[3] BL O'Dell, AR de Boland, SR Koirt-Yohann. Distribution of phytate and nutritionally important elements among the morphological components of cereal grains. Agricartueral and Food Chemistry, 1972,20: 718–722.

[4] J D Cook,S RLynch. The liability of iron deficiency:Blood, 1986, 68, 803–809.

[5] J D Cook,BSSkikne,RDBaynes, Iron Deficiency: The Global Perspective:*Advances in Experimental Medicine and Biology* , 1994, 356, 219–228.

[6] REBlack, Therapeutic and preventive effects of zinc on serious childhood infectious diseases in developing countries., American Journal of Clinical Nutrition, 1998, 68(suppl), 476S–479S.

[7] ZABhutta,SMBird,REBlack, KH Brown, JM Gardner, A Hidayat,F Khatun, RMartorell, NXNinh, MEPenny, JLRosado, SKRoy, MRuel, SSazawal, AShankar. Therapeutic effects of oral zinc in acute and persistent diarrhea in children in developing countries: Pooled analysis of randomized controlled trials:American Journal of Clinical Nutrition, 2000, 72, 1516–1522.

[8] TAhmad,R Bilal. Iron fortification of food: A strategy for control of iron deficiency anemia:The Nucleus, 2001, 38, 75–79.

[9] Recommendations on Wheat and Maize Flour Fortification Meeting Report: Interim Consensus Statement. World Health Organization. April 2009.

[10] ZAbdollahi, I Elmadfa, A Djazayery, MJ Golalipour, JSadighi, FSalehi, S Sadeghian Sharif. Efficacy of flour fortification with folic acid in women of childbearing age in Iran:Annals of Nutrition and Metabolism, 2011, 58, 188-196.

[11] CSJames.Analytical Chemistry of Foods., Blackie Academic and Professional, London. 1995.

[12] S Ranganna. Manual of Analysis of Fruits and Vegetable Products. Tata McGraw-Hill Pub., Company Ltd., New Delhi, 1978, pp: 109–110.

[13] R Nalubola, P Nestel.Manual For Wheat Flour Fortification With Iron; (Part3) Analytical methods for monitoring wheat flour fortification with iron, AACC Method 40-40. Iron-Qualitative Method, 1999, pp. 21-24.

[14] FDavidsin, RMiller.manual for wheat flour fortification with iron, analytical methods for monitoring wheat flour fortification with iron, 2005, 1-40.

[15] ANRao. Trace Element Estimation Methods and Clinical Context:Online Journal of Health and Allied Sciences, 2005, 4, 1-9.

[16] KA Riganakos, PG Veltsistas. Comparative spectrophotometric determination of the total iron content in various white and red Greek wines: Food Chemistry, 2003, 82, 637-643.

[17] JC De Andrade, FC Strong, NJ Martin. Rapid Determination Of Zinc And Iron In Foods By Flow-injection Analysis With Flame Atomic-absorption Spectrophotometry And Slurry Nebulization: Talanta., 1990, 37, 711-718.

[18] ZO Tesfaldet, JF Staden. equential injection spectrophotometric determination of iron as Fe (II) in multi-vitamin preparations using 1,10-phenanthroline as complexing agent: Talanta, 2004, 64, 1189-1195.

[19] J Head, B Getachew. *T*echnical Requirements for the Fortification of Wheat Flour in Ethiopia, Mekelle: Fulbright - Institute of International Education and Mekelle University, Mekelle,2014.

[20] AVogel.Textbook of Quantitative Inorganic Analysis.3rd Ed., Longmans and green and CO,New York, pp. 294, 310 and 787.