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Estimation of Iron Concentration in Different Brands of Wheat Flour Samples in Libya Markets, Using Spectrophotometer

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الملخص:

في هذه الدراسة تَحَقَّقَ من كمية الحديد في دقيق القمح لعدة الشركات، وأعتمدت هذه الطريقة علي تكوين معقد ملون بين الحديد الثنائي ومركب 10,1- فيننثرولين (phenanthroline-1,10). أجْرِيَ هذا التفاعل في وسط حامضي عند درجة حرارة 20° مئوية، وكان اكبر امتصاص للمعقد بين الحديد الثنائي ومركب 10,1- فيننثرولين (phenanthroline-1,10) عند طول موجي 510nm. وجدت علاقة خطية بين الامتصاص وتركيز الحديد يبدء من 0.2mg\kg الي 4 mg\k . نتيجة التحاليل لتحديد نسبة الحديد في عينات الدقيق كانت متفاوتة تبداء من 14.84mg\kg في شركة الزاد الطيب الي اعلي كمية وجدت في شركة زينا وكانت تقريبا حوالي 14.84mg|kg.وكانت ابتداء من نتائج متفاوته لشركات مثل رندا، دايري وشركة الصفوة،حيث وجدت النتائج كالتالي 30.3932.18 ,mg|kg و 35.22 علي التوالي، وبحسب المواصفات العالمية القياسية لكمية الحديد في الدقيق هي 33mg\kg ، وعلية فان النتائج التي تحصلنا عليها في شركات مثل رندا، دايري وشركة الصفوة كانت مقاربة للمواصفات العالمية القياسية لنسبة الحديد في الدقيق. وفي المقابل وجدت شركات اخري كانت فيها نسبة الحديد اقل من المعدلات المطلوبة مثل شركة السنبلة الذهبية، شركة موليو المونتا وشركة مصنع بنغازي والتي كانت كالتالي 23.59، 23.9 و 25.94mg/kg علي التوالي. وايضا اوضحت النتائج ان النسبة المئوية للرطوبة في قمح الدقيق لكل الشركات كانت ما بين 10.12% الي 12.83%، وهذا كان قريبا من النسبة المسموح بها وهي 12%. واضافة الى ذلك فإن النسبة المئوية للرماد في كل الشركات ما بين 0.38% الي 0.56%. وهو ايضا في المجال المسموح به وهو تقريبا 0.55%. اخيراء قيمة الأس الهيدروجيني PH في كل الشركات عامة ما بين 6.17 الي 6.41، وهذا ايضا يقع في المعدل الطبيعي للمواصفات العالمية القياسية وهي مابين 6 الي 6.8.

الكلمات المفتاحية:

تركيز الحديد، دقيق القمح، مقياس الطيف الضوئي، ليبيا.

Abstract

In this study, the measurement of iron content in various brands of wheat flours were investigated. The method was based on the formation of colored complex between Fe (II) and 1,10-phenanthroline. The reaction proceed quantitatively at 20°C in acidic medium. The maximum absorption of iron- 1,10-phenanthroline complex was at 510nm. A linear relationship was existed between the absorbance and Fe(II) concentration which is in the range of (0.2 to 4.0mg/kg). The analysis results of iron content in flour samples was variety, the average start from approximately 14.84mg/kg in Elzad Eltayb company, to the maximum value in Zina company roughly 47.90mg/kg. Relatively some companies results such as Randa, Diari and Alssafwa were convergent as 32.18, 30.39 and 35.22 mg/kg respectively. The standard agreement level of iron in wheat flour is 33mg/kg. Hence, our findings of these companies were in somewhat close the agreement level. On the other hand, the reading range obtained of Alsonbola Aldahbya, Molino Alimonti and Benghazi plain were around 23.59, 23.9 and 25.94mg/kg respectively. Also the results measurment of % moisture for the all companies brands of wheat flour were in the range of (10.12 to 12.83%), Which were around 12%, and this is the maximum allowed moisture level. In addition, % ash of all diverse companies were found to be from (0.38 to 0.56%). The ash content was in the limit allowed, approximately 0.55%. Finally, pH value of these companies were generally between (6.17-6.41), and that falls within the normal range of pH agreement of flour, (6.0 to 6.8).

Keywords: Wheat flour, Iron Concentration, Spectrophotometer, Libya.

1. INTRODUCTION

Wheat is one of the most widely produced grain crops in the world and is the basis for our global diet. To produce a viable food ingredient, most of the wheat was milled into white flour. Cereals are the main source of food in many countries, concerning human diet, and the most important cereals are wheat, rice, oats, barley, rye, corn and millet.[1] Among them, wheat is one of the most consumed and spread.[1] The wheat grain has the following average percentage composition (Endosperm 85%, bran 12.5% and germ 2.5%). The composition of wheat flour, however, varies considerably according to the class of wheat, its country of origin and the proportion of outer parts removed by the particular milling process.[2] The outer portions contain more portions, fat, fiber and ash than the starchy endosperm, the proportion of each of these constituents decreases as the extraction percentage gets less.[2]

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Iron is a vital element in our life. Iron is one of the mineral in human health, and playing an important role in immune function, cardiovascular health.[3] Iron deficiency occurs when iron requirements cannot be met by absorption from the diet, such as during periods of rapid growth (infancy, adolescence), in pregnancy, and as a result of menstrual or pathological blood loss.[4] Iron is available for supplement in two forms; heme and nonheme.[5] Heme iron is found in meat, fish and poultry. Heme iron is absorbed very efficiently in our body. Nonheme iron can be found in vegetables (spinach,corn) and beans (soyabeans, kidney beans), but its bioavaibility is poor. Absorption of heme iron is very efficient and not significantly affected by the composition of our diet. The meat protein and vitamin C can improve the absorption of nonheme iron.[5] However calcium, polyphenols and tannins, which are found in tea, rice and grains, could decrease the absorption of nonheme iron. Some protein found in soyabeans also inhibit nonheme iron absorption.[5] The orto-phenantroline forms with Fe (II) a stable red colored complex, which is called Ferrand.

Molar absorptivity of complex, [(C12H8N2)3Fe]2+, is 11,100 L mol-1cm-1 at 510nm.

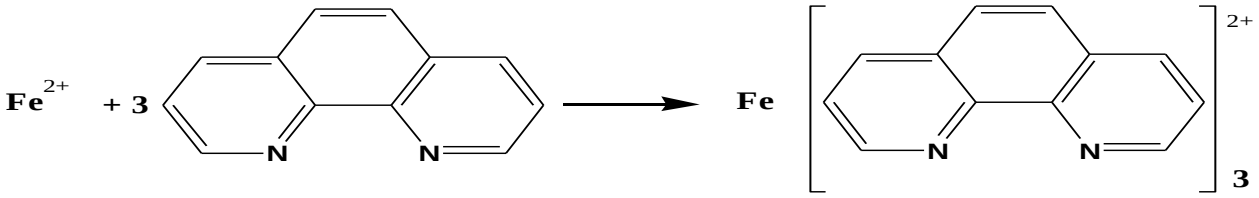

Figure 1. Reaction of 1,10-phenanthroline with Iron to form complex.

Based on this formation of complex, the main objective of this study was to investigate the moisture content, ash content and iron (Fe) content in wheat flour samples, which distributed under different brand names in Libya.

2. MATERIALS AND METHODS

The routine analysis of flour may include the determination of %moisture, %ash, added chalk ,sulfur dioxide, oil ,protein, acidity, iron, thiamine and nicotinic acid.

2.1. Material

All reagents used in this research were analytical-reagent and grade-pro analyze. Thus high-purity double distilled water was used for the preparation of all reagents and metal ion solution. Additionally, working solution of iron was prepared from standard stock solutions

whenever required. In general, the mixing of the flour sample is the most important one,

especially with fortified flour. Eight samples were collected from each brand and was weigh 250g each. The flour samples which were currently available for this study were thirty two in total. These studied were the estimation of iron content, pH, %moisture and %ash. The thirty two flour samples represented eight diverse brands as following: [Randa (Ra), Elzad Eltayb (Za), Diari (Farina) (Dr), Alssafwa (Farina) (Sf), Alsonbola Aldahbya (Sd, Molino Alimonti (Mo) (Farina), Zina (Zn) and Benghazi Plain (Bp)].

2.2.Methods

2.2.1. Determination of pH

To estimation of pH, Firstly, 10g of white flour sample were weighed and placed in an Erlenmeyer flask. Then added 100ml of distilled water at 25 ° C. The solution content was mixed for 30 min. After 30 min of mixing, the solution content was filtered and left to rest for 10 min, finally pH was measured using a pH meter.[6]

2.2.2. Determination of % moisture

A small sample of flour (2 to 3 grams) was weighed and placed in a crucible, then heated to 130 °C in an dry oven for 1 hour The sample then cooled to room temperature using desiccator. Moisture content was determined by heating a known weight of a flour sample in an air oven and comparing it with the weight of the sample after the heat to determine the amount of weight loss in the moisture content process. [7],

$$\% \text{ moisture} = 100 - [(W_{\text{drying}} / W_{\text{internal}}) \times 100]$$

2.2.3. Determination of % ash

A sample of flour which is weighted between (3 to 5 grams), was placed in porcelain crucible. The sample was heated to 600 ° C in the electric muffle furnace, until its weight was stable (usually overnight). The residue was then cooled to room temperature and weighed.[8] Ash content is determined by high temperature incineration in an electric muffle furnace. When the sample is incinerated in the oven, the high temperature drives out the moisture and burns away all the organic materials (starch, protein and oil), leaving only the ash. The residue (ash) is composed of the non-combustible, inorganic minerals that are concentrated in the bran layer.[9]

$$\% \% \text{ Ash} = [(W_{\text{ash}} / W_{\text{original sample}}) \times 100]$$

2.3. Preparation of the ash solution

In general, the official methods to determine minerals in flour is involve the complete digestion of the sample in a dry process or ashing.[10] However, The process is involving the addition of 5 ml of concentrated HNO_3 onto the inside walls of the crucible in order to dissolve the flour ash samples, add to the crucible. The acid then evaporate by heating the crucible on a hot plate at low temperature, the solution should not be boil. Also adding 2

ml of concentrated HCl to dissolve the remaining residue, then heat for few minutes , with taking an extra care that the solution dose not spill out the crucible . Let the crucible cool down, then the solution was filtered out and the filtrate was collected in 50ml volumetric flask. Distilled water was used to make up the volume, and was save for analysis.

2.4. Determination of standard curve of iron

Into a series of 50ml volumetric flask, added with pipit 0.0, 1.00, 2.50, 5.00, 7.50, 10.00, 15.00 and 20ml of standard iron solution. To each of the flasks added 1.00ml of hydroxylamine hydrochloride solution, then mixed them well and let each of them to stand for 5 minutes. Also to each of flask sample add 5.0ml acetate buffer and 5.0 ml of 1,10-phenanthroline. The samples should be mix well and the color will star to developing. Then let them stand for 30 min and then make up the volume to (50ml) using deionized water. The spectrophotometer turn on for 15-20 minutes, and the wavelength to 510 nm. Set the instrument to zero Absorbance using the blank solution. Then Read the absorbance of the standard solution and record the absorbance.

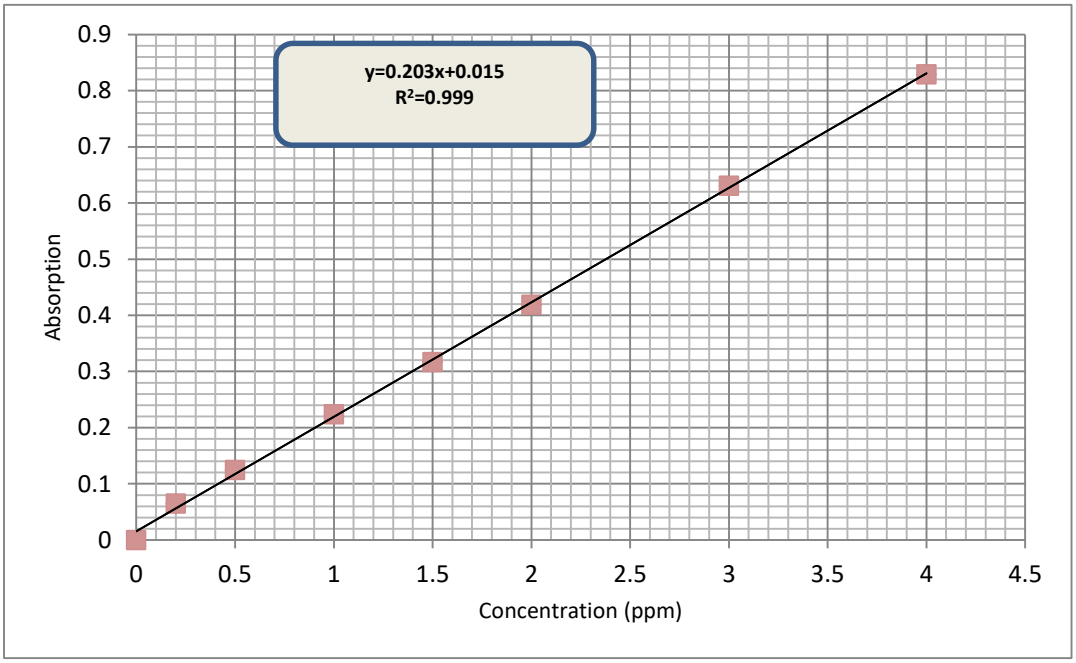

Figure 2. Calibration curve for determination of iron(II) in flour samples.

2.5 Instruments

Iron concentration were determination by double-beam spectrophotometer model CECIL (CE 7400, 7000SERIES). All absorbance measurements were made at 510nm of iron 1,10-phenanthroline complex with 1cm cell. Digital pH meter (mode pH 1741) was used to measure the PH of the flour solutions.

3. RESULTS AND DISCUSSION

The data related to amount of iron, %moisture and %ash in eight of wheat flour samples, which were presented in tables (**1** and **2**).

Table 1. Iron content (mg/kg) in different companies of white flour samples, (n=4) which determined by spectrophotometer.

Sample Code	Description of Sample	Concentration of iron mg/kg
Ra	Randa	32.18 ± 2.195
Za	Elzad Eltayb	14.84 ± 1.866
Dr	Diari (Farina)	30.39 ± 0.298
Sf	Alssafwa (Farina)	35.22 ± 0.589
Sd	Alsonbola Aldahbya	23.59 ± 0.313
Mo	Molino Alimonti (Farina)	23.99 ± 1.173
Zn	Zina	47.90 ± 0.638
Bp	Benghazi Plain	25.94 ± 1.432

S.D = Standard Deviation. p>0.05 between different brands of flour samples for all values

Table 2. Mean pH values, % Moisture and% Ash for commercially available different wheat flour samples in Libya.

Sample code	pH Mean ± SD	% Moisture Mean ± SD	% Ash Mean ± SD
Ra	6.17±0.078	11.73 ± 0.035	0.56 ± 0.120
Za	6.24±0.049	10.20 ± 0.000	0.52 ± 0.035
Dr	6.19±0.000	12.33 ± 0.035	0.38 ± 0.021
Sf	6.19±0.021	12.40 ± 0.283	0.51 ± 0.042
Sd	6.17±0.120	10.47 ± 0.177	0.44 ± 0.014
Mo	6.41±0.410	11.67 ± 0.445	0.40 ± 0.014
Zn	6.34±0.141	12.83 ± 0.106	0.49 ± 0.021
Bp	6.41±0.000	11.65 ± 0.000	0.50 ± 0.016

First of all, noticed that in table (1) maximum concentration of white flour samples of Zina brand exhibited the maximum value of iron, which is found to be 47.90 mg/kg. but the amount of iron decreased gradually to approximate 14.84mg/kg as shown in Elzad Eltayb company. As we can see in Alsonbola Aldahbya and Molino Alimonti, the range of Fe content varied from 23.59 to 23.99 mg/kg respectively. Also the amount of iron in Benghazi plain company was adjacent to be 25.94 mg/kg, whereas the amount of iron in Randa and Diari companies were slightly above average of values of iron of the other company, which were about 32.18 and 30.39 mg/kg respectively. In general, table (1) illustrated the concentration of iron in different brands. Accordingly to SPSS which was used to different types of wheat flour samples, In the case of Fe, all various brands of samples were found to have a significant variation of Iron.

As it observed, in this study that the obtained values of Fe in marketed mill flour samples, which contained a low values of iron indicating deficiency of the element. It could be explained that minerals are always found in the grains, especially in the germ and outer layer of kernel and milling process is responsible of removing the germ and most of the outer layer of the kernel produces white flour, which has a higher proportion of starch and higher caloric value than the whole wheat flour and some of the vitamin and mineral contents of the kernel are always lost in milling white flours.[15] The standard level of iron in wheat flour is 33mg/kg.[8,16] Hence, our findings were pretty close to the studies reported earlier.

Table 3. Comparison of the obtained results for Iron element with other author's results (mg/kg fresh weight).

Origin	Fe (mg/kg)	Reference (year)
Italy	36.9 – 38.0	Locatelli, 2014.[11]
India	43.8	Singh & Gary, 2006. [12]
China	5.5 – 15.7	Tang et al, 2008.[13]
Spain	8.17 – 8.63	Raquel et al, 2013.[1]
Pakistan	16.0 - 49	Ihsanullah et al, 2002.[8]
Hungary	6.36 – 12.46	Szira et al, 2013.[14]
Libya	14.84 – 47.90	Current study, 2019

According to table (3.3), which revealed the comparison between the results of the analyzed iron element in the flour samples with spectrophotometer, which obtained in this study and those of other authors were agreement together. On the other hand, the results obtained of Fe content were good quality of wheat flour samples, specially of Randa, Diari, Alssafwa and Zina comparison with standard level. The data showed in table (3.2), indicated that the % moisture was quite low in the whole wheat flour samples. Accurate measurement of moisture content is very difficult. The water in food items exists in three different forms i.e. bound form (water of crystallization or as hydrate), adsorbed water (physically bound as a monolayer to the surface of the food constituents) and bulk or free water (separate constituents). The free water is lost by evaporation. However, the maximum allowed moisture level is 12%. Moisture values include other volatile matter such as essential oils, traces of volatile acids and amines.[8] Moisture flour >13% is liable to attack by microorganisms, mites and insects. Also highest moisture content is mainly responsible for microbial spoilage and hence we can't store flour for long time. Consequently from the standpoint of view, the moisture content of the flour samples were in a pretty good values, which are well below the

maximum limit. In addition, the ash content was in the allowed limit that is approximately 0.55%.[9] Also pH value of this companies were generally (6.17-6.41), and that usually falls within the range of pH of flour (6.0-6.8).[2]

4. CONCLUSION

The use of spectrophotometer method with 1,10-phenanthroline mono hydrate reagent can be successfully applied to the determination of iron in wheat flour. Moreover, the ortho-phenanthroline spectrophotometer methods were found to give satisfactory results. Also the orange-red color complex formed was stable for a number of hours. Finally, this procedure was selective for Fe^{2+}, even in the presence of other metals.

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