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Spectrophotometric determination of iron in dietary Supplements in Libyan market

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Abstract:

In most cases dietary supplements especially in Libya are consumed without prescriptions and the public has very limited any awareness of their health risk. The lack of quality control on numerous brands of dietary supplements in pharmacies of Libya today may result in a serious health problem.

The aim of this study is to determine the iron content using a fast and accurate method for quality control of some imported dietary supplements, based on spectrophotometric measurement of iron after complexation with 1, 10-phenanthroline in an acidic medium. Eight types of vitamin supplementary tablets were randomly collected from the Libyan market and analyzed for the iron content. The analysis showed an average value of 61mg Fe/pill for the range of 40.07-112.63 mg Fe/pill. Results showed that 75% of the samples were lower in iron content than that recorded on the dietary products.

Keywords: Dietary Supplement; Spectrophotometer; Iron content; Libyan Market.

1- Introduction

There are two main forms of iron salts with numerous formulations such as: amino acid chelates, carbonyl iron, polysaccharide iron, combination products and extended release products available globally. Since ferrous iron is absorbed three times more readily than the ferric form, all nutritional iron has to be reduced to the ferrous form (McEvoy, 2007). In food, there are two types of iron: heme and non-heme iron. Heme iron is easily absorbed and used in our body and is mainly found in meat. Whereas non-heme iron is mainly found in plants (Dubey et al., 2015). Although, historical claims state that sustained-release iron preparations caused fewer gastrointestinal side effects yet have not been well established (Boliger & Korma, 1990).

Whereas, there is some evidence that controlled-release iron preparation causes less nausea and epigastric pain than conservative ferrous sulfate, but the discontinuation rates are similar (McDiarmid & Johnson, 2002).

However, it has been reported that iron absorption may be improved by the addition of vitamin C to some supplements. About 200mg of vitamin C is required to increase absorption of 30 mg of elemental iron (McEvoy, 2007). Most iron supplements containing vitamin C do not have an appropriate amount of vitamin C to significantly affect iron absorption, therefore it is better if iron supplements are taken on an empty stomach since food can decrease iron absorption by 40-50% (McEvoy, 2007; Boliger & Korma, 1990).

The main iron salts used as iron supplements are: ferric ammonium citrate, ferrous bisglycinate, ferrous fumarate, ferrous gluconate, ferrous sulfate, folysaccharide-iron complex (Nagpal & Choudhury, 2004).

The World health organization WHO reported that about 42% of pregnant women are anemic worldwide. At least half of this anemia burden is assumed to be due to iron deficiency (WHO, 2012).

A pregnant woman is considered to be anemic if her hemoglobin concentration during the first and third trimester of gestation is lower than 11.0 g/dL (WHO, 2011). When anemia is accompanied by an indication of iron deficiency (e.g. low ferritin levels), it is referred as iron deficiency anemia (WHO. 2001).

It is customarily necessary to analyze a specific chemical species in the presence of a number of others.

When the species of interest can be made to have a unique color, spectrophotometric analysis can be an easy, quick and accurate method. Standard methods are based on converting iron (III)

to iron (II) using reducing agents such as hydroxylamine or hydroquinone to develop a reddish orange complex with o-phenanthroline (Elmagirbi et al.,2012). In this work we conclude that quality control is important in the assessment of dietary supplements for the safety of anaemic patients, using a fast spectrophotometric method. However, a recent redox titration method has been reported for the determination of iron in iron containing tablets (Balarabe and Folashade, 2019).

2- Practical work

2-1- Materials

All chemicals used in this research were of high purity and deionized water used, 0.2 % of 1,10-phanathroline (Aldrich 99%), 6 M hydrochloric acid (Aldrich 37%), 10 % sodium acetate (BDH, AnalaR), 10 % hydroxylamine hydrochloride (Chem service inc), ferrous ammonium sulfate hexahydrate (BDH).

2-2- Procedure

Eight types of supplementary tablets (iron or iron-folic acid base) were purchased from the Libyan market; ten tablets of each type were weighed individually to calculate the average weight of one tablet. The tablets were ground manually using an agate Mortar and pestle. Digestion was performed similar to that reported (Atkins 1975). To prepare the stock solution (A), in three replicates 0.10 g of the crushed tablets were placed in 150mL beaker, boiled gently in a fume hood with 25mL of 6M HCl for about 15 minutes while covered with a watch glass. Deionized water was added when necessary to keep a constant volume (15mL). Finally the watch glass was rinsed with water, and the solutions were then filtered directly through Whatman-40 filter paper receiving the filtrates in 250mL volumetric flasks. The remaining precipitate was rinsed thoroughly with deionized water and completed to the mark with deionized water. For the measuring solution (B), to a 100mL volumetric flasks, 2mL of the stock solutions (A) were transferred and the reagents were added in the following amounts: 10.0mL of 10% hydroxylamine hydrochloride, 10.0mL of 0.20% 1,10-phenanthroline and 8.0mL of 10% sodium acetate.

2-3- Standard solutions

A 10mg/L Fe standard solution was prepared by accurately dissolving 0.0700g of pure ammonium ferrous sulfate $(NH_4)_2Fe(SO_4)_2.6H_2O$ in 1000mL volumetric flask using deionized water. From the standard solution, 0, 5, 10, 25 and 50mL aliquots were transferred each to 100mL volumetric flasks. The same amounts of the reagents added to the measuring solutions of the samples were added to the standard flasks to give a series of calibration solutions with Fe concentrations of: 0, 0.5, 1.0, 2.5 and 5.0mg/L respectively. The 5.0mg/L Fe solution was used to obtain λ_{max} which was found to be 510nm when fully scanned from 200 to 800nm Using GBC-Cintra 2020 UV-Visible spectrophotometer.

The calibration solutions were then measured quantitatively to gain the absorbance value for each solution. A calibration curve was then established showing a relation between the concentration (mg/L) and the absorbance using the excel program. The samples were measured at the same conditions and their concentrations (C_i) were calculated from (equation 1) obtained from the calibration curve (figure 3.1). Finally, the iron concentration of each sample in mg/pill was then calculated from equation 2, which has been designed according to the dilution factor, the total volume and the sample weight.

$$Y = 0.2033\ C_i \qquad \text{(Equation 1)}$$

Where:-

Y = absorbance and C_i is the Fe concentration of each sample solution (mg/L).

$$Fe(mg/pill) = \frac{C_i \times 100 \times 0.250 \times pill\ weight(g)}{2 \times sample\ weight(g)} \qquad \text{(Equation 2)}$$

Where:-

Pill weight = average of 10 tables and

Sample weight = weight of the crushed portion that was dissolved.

3- Results and discussion

3-1 Calibration curve

Figure 3.1 shows the calibration curve for iron concentration (mg/L) against the absorbance measured at 510nm. A linear relationship was obtained with a simple equation $Y = 0.2033C_i$, $R^2 = 0.9971$ and LOD is 0.21mg/L.

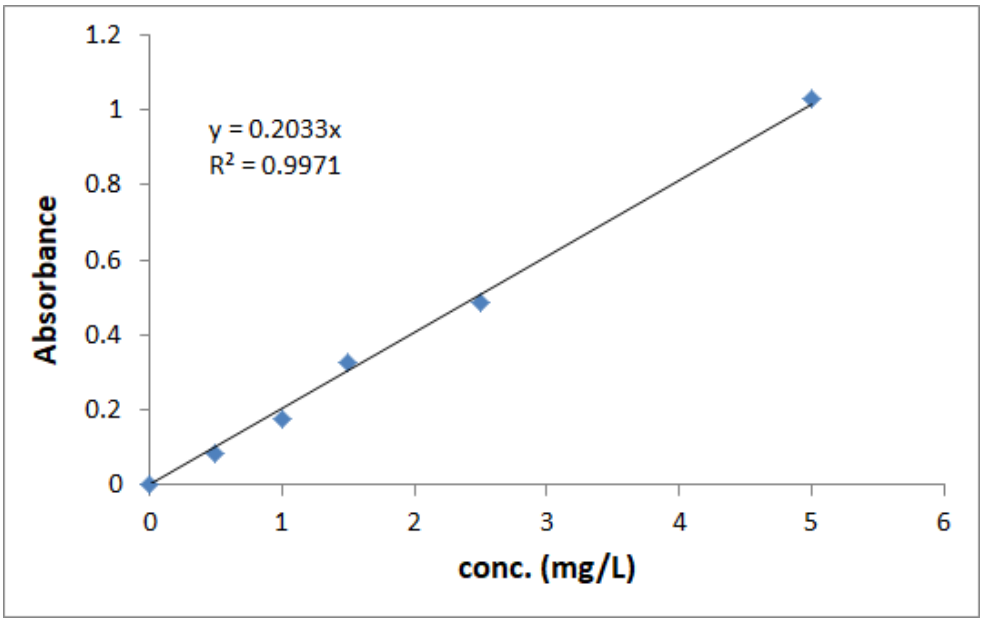

Figure 3.1 UV-Visible Calibration curve for Fe standard solutions

3-2- Recovery percent

Method validation was implemented by running standard solutions as samples and the recovery percent was obtained as shown in Table 3.2. From the obtained values one can see that the UV/Vis spectrophotometer is a suitable technique to measure the iron content in medicine tablets.

Table 3.2 method recovery percent

STD soln.	Theoretical value	Measured value	Recovery percent
STD 1	0.50	0.42	84%
STD 3	1.50	1.67	111.3%
STD 4	2.50	2.56	102.4%
STD 5	5.00	4.29	85.8%
Overall average			95.88%

3-3 Supplements iron content

The iron content in the analyzed dietary supplements differs from one to another. The resulting concentrations ranged from 40.08mg/pill to 112.63 mg/pill showing an average amount of 60mg/pill iron, while the median was 50.6mg/pill. There is a significant difference between the iron content recorded on the supplement pack and the actual measured value in almost all samples, apart from samples S4, S6 and S8 were compatible. A comparison of the obtained result (actual) and those recorded on the tablet container are shown in figure 3.2.

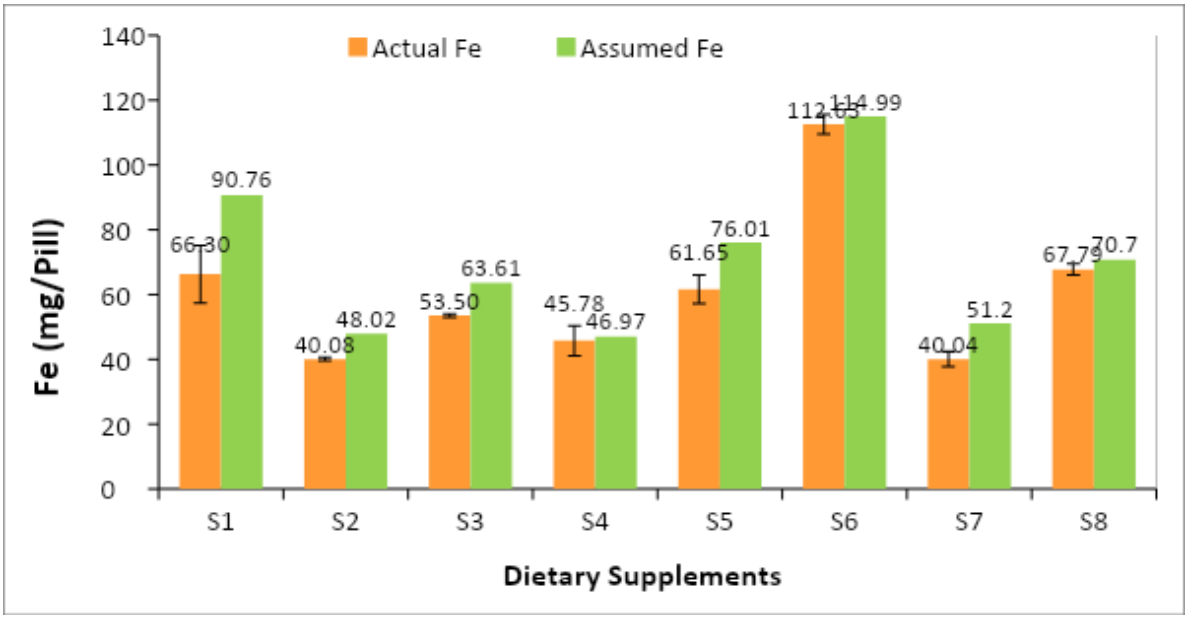

Figure 3.3 Comparison between assumed and actual Fe content (mg/pill).

The samples S1, S2, S3, S4, S5, S6, S7 & S8 showed a % difference of 36.9, 19.8, 18.9, 2.6, 23.3, 2.1, 27.9 and 4.3 respectively.

4- Conclusion

The analysis indicates that the dosage of iron inscribed on the container of the medicine is not equivalent to that measured. This difference may lead to serious health problems especially in anemic pregnant women. According to the WHO guideline report (WHO 2012), pregnant women which are clinically diagnosed as anemic should be initially cured with a higher daily dose of elemental iron (120mg) supplementation until the normal hemoglobin concentration is reached before switching to the standard antenatal dose to prevent repetition of anemia. Therefore, routine analysis of iron in dietary supplements is necessary to ensure that the correct amount of iron is provided.

Accordingly, the authority must take on advance the responsibility for the quality control of any imported medicine before reaching patients. Since there are serious side effects related to the iron content such as anemia, lethargy, heart palpitations and tinnitus low levels of iron while overdosing may be associated with appreciable morbidity and mortality cases. Multi system failure is common in lethal overdoses of iron (Gerald 1994).

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