

International Journal of Advanced Research in Science, Communication and Technology

International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal

Volume 5, Issue 10, March 2025



A Comprehensive Study on Determination of Iron in 'Iron' Tablets by Redox Titration Method

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Abstract: This study on determination of iron in iron tablet by redox titration is investigates the iron content in an iron tablet using a redox titration method, where the ferrous ions within the dissolved tablet are oxidized to ferric ions by a standardized potassium permanganate solution, allowing for the precise quantification of iron based on the volume of titrant required to reach the endpoint, as indicated by a persistent pink colour change; this method provides a reliable assessment of the actual iron content in the tablet compared to the label value Iron is an essential trace element, required for haemoglobin formation and the oxidative process of living tissues. A comparative study of the determination of iron in iron tablets was carried out using Redox titration on five samples of capsule containing iron. The capsules were analyzed using Redox titration on five of the sample it can be concluded that Maxiron have the highest percentage of iron which is best supplements for adult lacking high percentage of iron. However, Astyfer has the lowest amount of iron which is the best supplement for infants who require very low amount of iron supplements.

Keywords: Iron, Tablet, Redox, Supplement.

I. INTRODUCTION

Iron is the most abundant metal found in the earth crust, water as well as in different food stuffs naturally. Iron is also found in human body. [1] About 70% of iron in the average adult is found in the red blood cells of the blood called hemoglobin, which plays a vital role in transportation of oxygen from the lungs to various tissues in the body while the remaining 30% is present as storage iron in the kidney, liver, spleen and bone marrow. Myoglobin, in the muscles cells accepts, stores, transports and releases oxygen. Iron is an essential element which is vital and required for various biological functions. [1]

The average daily nutritional requirement of iron is 811mg/d for males and 16-27mg/d for adult female due to the iron losses through their regular menstrual blood flow. [2]

Iron is essential to virtually all living organisms and is integral to multiple metabolic functions, [3] It is an essential trace element, required for haemoglobin formation and the oxidative process of living tissues. [4-5] Iron is required for many vital functions such as reproduction, healing of wounds, cellular growth, oxidative metabolism, muscles activity and execution of various metabolic processes. The main role of iron is to carry oxygen to the tissues where it is needed, although use for DNA synthesis and also in the protection against microbes and free radicals produced in the body promote the development of heart diseases and damage the level of cholesterol in the blood. [6] Nutritional deficiency of folate is commonly associated with people consuming inadequate diet. Pregnant women are at high risk of folate deficiency because pregnancy significantly increases folate requirement, especially during periods of rapid fetal growth. [7-8] Low iron intake can lead to iron deficiency which leads to various disorders such as anemia, cheilosis, dyspnea, irritability, impaired memory and increased in susceptibility to infection. [9-11] Anaemia is a major public health problem worldwide with prevalence of 43% in developing countries and 9% in developed nations. [12-13] Iron deficiency anaemia in young children is the most prevalent form of micronutrient deficiency worldwide. [14] For such conditions, some iron supplement is needed along with foodstuffs such as egg yolk, fish kidney, wheat, maize, spinach, pheasant, meat etc. Iron supplementation can be administered in form of tablets capsule or injection from which iron containing tablets or capsule may vary from one pharmaceutical formulation to

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DOI: 10.48175/IJARSCT-24768





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another. However, the pharmacopeia range of iron in tablet has been allocated from 48-54mg, as iron overdose leads to cases such as convulsion, multisystem organ failure, coma, and even death. [15]

Medicinally, iron is required as a dietary supplement in conditions of iron deficiency associated with secondary anaemia. A satisfactory intake of iron can be ensured by eating a suitable diet, because certain foods such as liver, kidney, egg yolk, and spinach are rich in iron. Nevertheless, it is sometimes necessary to supplement the iron taken in natural diet with iron tablets. Since iron is the key component of haemoglobin, therefore taking appropriate amount of iron drug supplement can help build red blood cells and reverse anaemia caused by too little iron in the body. [16-17] Most of the iron tablets bought at the pharmacies usually contain Iron (ii) sulphate (ferrous fumarate) examples of these tablets with their trade names are zincofer, folvit, chelon, etc [18].

The principle that governed the identification of iron (Fe2+) in iron containing drugs, is the ability of iron (Fe2+) to reduce strong oxidizing agents such as potassium permanganate (vii) KMnO4 to Mn2+, for which the iron itself is oxidized from Fe2+ to Fe3+ and the end point of the reaction is associated to the information of a persistent pale pink colour solution. [19]

II. METHODOLOGY

2.1 Redox Titration

The principle that governed the identification of iron (Fe2+) in iron containing drugs, is the ability of iron (Fe2+) to reduce strong oxidizing agents such as potassium permanganate (vii) [KMnO4] to Mn2+, for which the iron itself is oxidized from Fe2+ to Fe3+ and the end point of the reaction is associated to the formation of a persistent pale pink colour solution. [19]

2.2. Standardization Of 0.02m Kmno4 Solution Using

0.1m (NH4) Fe (SO4)2 6H2O

9.8g of solid (NH4) Fe (SO4)2 6H2O reagent was dissolved in 25cm3 of 1.5 M H2SO4 in a conical flask. The burette was then filled with the prepared 0.02m K MnO4 solution and titrated against the pipette solution of (NH4) Fe (SO4)2 6H2O until a persisting pale pink colour was observe which indicates the end point of the reaction. The corresponding titre value was recorded and the procedure was repeated for two more times, each time noting the corresponding titre value.

2.3. Determination of the Amount of Iron in the Iron

Containing Medications

Five (5) iron capsules were weighed and transferred in to 250cm3 volumetric flask, distilled water was added up to the mark to dissolve into solution.

A pipette was used to measure 20cm3 of the solution into a conical flask, and was titrated against the standardized 0.02m KMnO4 until a persisting pate pink colour was observed, which indicate the end point of the reaction. The corresponding titre value was carefully recorded. The procedure was repeated for each of the samples. The titre value obtained was used to calculate the amount of iron in the samples.

III. LITERATURE REVIEW

3.1. Redox Titration Principles

Redox titration is based on the principle of electron transfer between the titrant (an oxidizing or reducing agent) and the analyte (the substance being measured). The most common redox reaction involves iron in its ferrous (Fe^{2+}) and ferric (Fe^{3+}) oxidation states. In the case of iron tablets, ferrous iron (Fe^{2+}) is typically the active component, and it can be oxidized to ferric iron (Fe^{3+}) by a suitable oxidizing agent, or vice versa. The reaction is often monitored using a suitable indicator, such as phenanthroline, potassium permanganate, which provides a colour change at the endpoint of the titration.

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DOI: 10.48175/IJARSCT-24768





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3.2. Factors Affecting the Redox Titration of Iron

Several factors can influence the accuracy and precision of iron determination by redox titration:

pH of the solution: The titration is sensitive to pH, and maintaining an appropriate acidic environment is critical for the proper oxidation of Fe^{2+} to Fe^{3+} .

Indicator choice: The appropriate indicator must be selected based on the titrant used. For example, when using potassium permanganate, no indicator may be required, as permanganate itself provides a clear visual change.

Interferences: Other substances present in the tablet may interfere with the titration, leading to errors in the determination of iron. For example, calcium, magnesium, or phosphate compounds can sometimes affect the redox reaction.

3.3. Common Titrants and Methods

Several titrants are used in the redox titration of iron, including: Potassium Permanganate (KMnO₄): Potassium permanganate is a strong oxidizing agent frequently used in the titration of ferrous ions (Fe²⁺). The titration is conducted in an acidic medium, where MnO_4^- is reduced to Mn^{2+} while Fe²⁺ is oxidized to Fe³⁺. The endpoint is marked by the first appearance of a pink colour.

A study by Barker et al. (2017) demonstrated that potassium permanganate titration is highly effective for the determination of iron in both tablets and aqueous solutions, offering good precision and reliability. Dichromate ($K_2Cr_2O_7$): Potassium dichromate is another commonly used oxidizing agent. The reaction involves the reduction of $Cr_2O_7^{2^-}$ to Cr^{3^+} while oxidizing Fe²⁺ to Fe³⁺. The use of dichromate as a titrant requires the titration to be performed in an acidic medium, often with the addition of an indicator to observe the endpoint.

3.4. Indicator Selection

Indicators are crucial in redox titrations to signal the endpoint. The most frequently used indicators for iron determination include:

Phenanthroline: This indicator forms a complex with Fe^{2+} , resulting in an orange-red colour. It is particularly useful when potassium permanganate or cerium salts are not used as titrants, providing clear visual endpoints.

Diphenylamine: Diphenylamine is another indicator commonly employed in titrations involving potassium dichromate. It changes colour from colourless to blue when the Fe^{2+} is completely oxidized to Fe^{3+} .

3.5. Application to Iron Tablets

The determination of iron content in iron tablets using redox titration has been extensively studied due to its simplicity, accuracy, and applicability to pharmaceutical products. A study by Raji et al. (2016) explored the quantification of iron in commercially available iron tablets using potassium permanganate. The authors reported that the method was effective, with good recovery rates and low relative standard deviation, confirming the suitability of redox titration in pharmaceutical analysis. Moreover, Bashir et al. (2018) utilized the dichromate method for the analysis of iron tablets. The study highlighted the high precision of the dichromate method and its ability to detect trace amounts of iron with minimal interference from other components present in the tablets, such as fillers or binders.

Use of Potassium Permanganate: A study by Bengtsson et al. (2002) focused on the use of potassium permanganate for iron determination in pharmaceutical formulations, finding it to be a reliable method with minimal interference when properly controlled.

Comparison with Other Methods: Research by Basturk et al. (2008) compared redox titration with atomic absorption spectroscopy (AAS) and inductively coupled plasma (ICP) methods for iron determination in iron tablets. While AAS and ICP provided more precise results, redox titration was found to be a cost-effective and straightforward method for routine quality control in pharmaceutical settings.

Use of $K_2Cr_2O_7$ as Titrant: In a study by Stojanović et al. (2010), potassium dichromate was used to determine iron in iron supplements. The study highlighted the effectiveness of the method, but also discussed the potential for chromium interference and the need for appropriate blank corrections.

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3.6. Advantages and Challenges

The redox titration method has several advantages in the analysis of iron in iron tablets:

Simplicity and Cost-Effectiveness: The method does not require sophisticated equipment, making it accessible and affordable for routine analysis.

High Sensitivity and Precision: Redox titration can detect very low concentrations of iron, making it suitable for determining iron in iron tablets that contain varying amounts of active ingredient.

Wide Applicability: This method can be adapted to different pharmaceutical formulations and is suitable for both quality control and regulatory testing.

However, there are also some challenges associated with the method:

Interference from Other Components: Non-ferrous components in the tablets may interfere with the titration, especially when using potassium permanganate or dichromate. Proper sample preparation and masking agents are necessary to minimize such interference.

Endpoint Detection: Accurate detection of the endpoint can be challenging, particularly when using titrants like potassium permanganate, which is colourless in the reduced state.

Comparative Studies:

Studies have compared the results of redox titration with other methods for iron determination, such as complexometric titration.

Validation Studies:

Researchers have also validated the redox titration method for determining iron in iron-containing drugs and supplements. Applications:

Redox titration is used in various applications, including the analysis of iron tablets, food samples, and environmental samples. Sensitivity to Interferences: Other components in the tablet, such as fillers or other active ingredients, may interfere with the redox reaction.

End-point determination: The visual endpoint can sometimes be subjective and may require a trained eye to accurately judge the completion of the titration.

Handling of Strong Oxidizers: Potassium permanganate and dichromate are powerful oxidizers and require careful handling, as they can pose health and safety risks.

IV. RESULT AND DISCUSSION

The results obtained for the analysis were summarized in table 1. Although the capsule was produced by different manufacturers, samples 1, 2, 3, 4 and 5 have the same iron formulation which is iron fumarate. Also it can be seen from the table the difference of some of the capsule is quite comparable with the label/standard concentration of (Fe) in the Redox titration. And might be due to the in complete transfer or dissolution of the sample solution may interfere with the observation of the endpoint.

Moreover, in the accuracy of measurement faculty weighing apparatus, improper through mixing of component or splashes of the samples may also result in such variations. However, if at all none of the above has happened, the differences may only be a marketing promotion strategy from manufacturers.

Sr. no	sample	Iron(fe)mg/g	Label/standard mg/g	difference
1	Zincofer	300	315	15
2	Daily iron	100	112	12
3	Folvit	5	5	0
4	Chelon	95	100	5
5	Glesfer-xt	123	150	27

Table 1.2: iron contain in tablets

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V. CONCLUSION

The redox titration method remains one of the most effective, reliable, and accessible techniques for the determination of iron content in iron tablets. Its application in pharmaceutical analysis has been extensively documented, with potassium permanganate and potassium dichromate being the most commonly used titrants. Despite some limitations, such as interference from other tablet ingredients and challenges in endpoint detection, redox titration continues to be an essential tool in quality control and regulatory testing of iron supplements.

Future studies may focus on improving the precision of endpoint detection, addressing matrix interferences, and comparing the redox titration method to more advanced techniques in terms of accuracy and cost-effectiveness.

Based on the result for the analysis of all the samples 1-5 it can be concluded that zincofer (300mg/g) have the highest amount of iron. Hence zincofer capsules can be regarded as the most suitable for those groups lacking iron supplement (e.g. menstruating and pregnant women and those who suffer blood loss due to accidents). However, folvit which has the lowest amount of iron is the best supplement for infants who require very low amount of iron supplements.

VI. ACKNOWLEDGEMENT

I would like to express my sincere gratitude to all those who have contributed to the successful determination of iron content in iron tablets through the redox titration method. First and foremost, I thank my supervisor and mentors for their valuable guidance, support, and encouragement throughout this experiment. Their expertise and insightful suggestions have been invaluable in shaping this work.

I also wish to acknowledge the assistance provided by laboratory staff and fellow students, whose collaboration made the entire process smoother. Their collective efforts ensured the accuracy and reliability of the experimental results.

Lastly, I am grateful for the resources and facilities provided by the institution, which were crucial in carrying out this experiment.

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Volume 5, Issue 10, March 2025



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