International Journal of Applied Mathematics, Sciences, and Technology for National Defense
Volume 1, Issue 2, 51-58.
e_ISSN: 2985-9352, p_ISSN: 2986-0776
https://www.journal.foundae.com/index.php/JAS-ND
DOI : https://doi.org/10.58524/app.sci.def.v1i2.165

Iron determination in multivitamin tablets: Enhancing military nutritional preparedness based linear regression method

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Article Info

Article history:

Received: April 27, 2022
Revised: July 11, 2023
Accepted: August 05, 2023
Published: August 30, 2023

Keywords:

AAS
Iron
Linear Regression
Sensitivity

Abstract

AAS is also an important method that has the most extensive application in metal analysis. most of the determinations were carried out by thin spectrophotometry, therefore an experiment was carried out to determine the Fe content in multivitamin tablets using the atomic absorption spectrophotometry method has high selectivity and sensitivity. Standard solution preparation. Prepare a standard solution of Fe with a concentration of 1, 2, 3, 4, and 5 ppm, then read the absorbance of the standard solution on AAS. Sample solution preparation and absorbance measurement. Several tablets were put into a beaker and 7 ml of concentrated HCl was added working principle of atomic absorption spectrophotometry is based on the evaporation of the sample solution which will initially be nebulized to form a spray, then it will be desolvated to form a dry aerosol until it is evaporated and converted into free atoms. Iron levels in samples using AAS can be found by measuring the intensity of the radiation that decreases after being transmitted. The reduction in radiation intensity is proportional to the concentration of the sample element in the sample being measured. By testing the t-test, it was found that the AAS instrument was more sensitive to low concentrations

To cite this article: Prasojo, E. A., Anitasari, R., Sijabat, W. N., & Nurrosyid, N. (2023). Iron determination in multivitamin tablets: Enhancing military nutritional preparedness based linear regression method. *International Journal of Applied Mathematics, Sciences, and Technology for National Defense. 1*(2), 51-58.

INTRODUCTION

Fe analysis plays a key role in ensuring the quality, reliability, and safety of iron-based materials in the defense sector. With the help of this analysis, appropriate steps can be taken to maintain and increase the readiness of the country's defense technology. Besides that, Iron is one of the more important elements in surface water and groundwater (Heikkinen et al., 2022). This is one of the microelements needed by the body. It has many roles in the body's metabolic processes (Gupta, 2014). This metal will become toxic if it is present in concentrations above normal resulting in damage to important organs such as the pancreas, heart, muscle, and kidneys (Supriyantini & Endrawati, 2015). Iron deficiency in the human body can be avoided by providing adequate iron intake. Treatment of anemia is not enough just by changing food consumption but can also be overcome by taking blood booster tablets must be by a predetermined dose so that the iron contained in the body is not excessive (Miller, 2013). Anemia sufferers must consume blood booster tablets with iron levels of 60 mg 1-2 times a day (Sugiarso & Kurniawati, 2016). Before circulating in the

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Multivitamin Tablets market, its quality must first be tested. This must be done to ensure that the Multivitamin Tablets contains the correct ingredients with the menu and that the specified amount is made under constant conditions and following the applicable standard procedures so that the Multivitamin Tablets meets the established specifications for levels and safety. The choice of method is a very important issue in all analyses because the proper separation of methods will require accurate analysis results and minimize analysis errors.

One of the methods that can be used to determine the Fe (II) content is the atomic absorption spectrophotometry method (Bağ et al., 2001; Brahmana et al., 2020). Atomic absorption spectrophotometry (AAS) has the principle of neutral atoms being excited in the ground state and absorbing radiation from a light source with a certain wavelength (Madania & Martani, 2014). The light source in AAS is a light source from a cathode lamp that comes from the element being measured and then passed into a flame containing an atomized sample, then the radiation is passed to the detector (monochromator). The AAS method has high selectivity, limits detection which has a small error of about 0.5 to 2% under optimum conditions, and is relatively free from AAS interference. It is also an important method that has the most extensive application in metal analysis. most of the determinations were carried out by thin spectrophotometry, therefore an experiment was carried out to determine the Fe content in Multivitamin tablets using the atomic absorption spectrophotometry method has high selectivity and sensitivity.

METHOD

Tools and Materials

The tools used were Atomic Absorption Spectrophotometer, 50 ml measuring flask, Fe Cavity Cathode lamp, funnel, 10 ml Mohr pipette, 50 ml beaker, 10 ml measuring cup. The materials used were concentrated HCl, 100 ppm Fe standard solution, deionized water, multivitamin tablets, and filter paper.

Procedure

Standard solution preparation (see Table 1). Prepare a standard solution of Fe with a concentration of 1, 2, 3, 4, and 5 ppm, then read the absorbance of the standard solution on AAS. Sample solution preparation and absorbance measurement(Alizah et al., 2019). A number of tablets were put into a beaker and 7 ml of concentrated HCl was added, then heated slowly using a warm plate in an acid chamber until dissolved (black like charcoal) (Alizah et al., 2019). After that, add 50 ml of deionized water and filter with Whatman paper No 40 when put into a 100 ml measuring flask(Alizah et al., 2019). Wash the residue with 25 ml of deionized water and calibrate(Alizah et al., 2019). Sample preparation was carried out in triplicate. Read the absorbance of the sample solution on AAS as show in Table 2. Data Analysis. Construct a calibration curve showing the signal of the Fe standard solution (see Figure 1). A linear regression line is then constructed to determine the slope and intersection of the curves. Y is the atomic absorption signal and X is the concentration of Fe in ppm. Calculate the Fe content in multivitamin tablets (% w/w) along with the t test and 95% confidence interval. The Fe content can be calculated by substituting the absorbance value (Y) from the results of the analysis on AAS, after which the Fe concentration will be obtained which can be converted to Fe content (% w/w)

Table 1. Absorption Fe (III) Standard Solution

Standard Solution	Volume Standard	[Standard] (ppm)	Absorbance
0	0	0	-0.0031
1	2.5	1	0.0037
2	5	2	0.0121
3	7.5	3	0.0223
4	10	4	0.0275
5	12.5	5	0.033

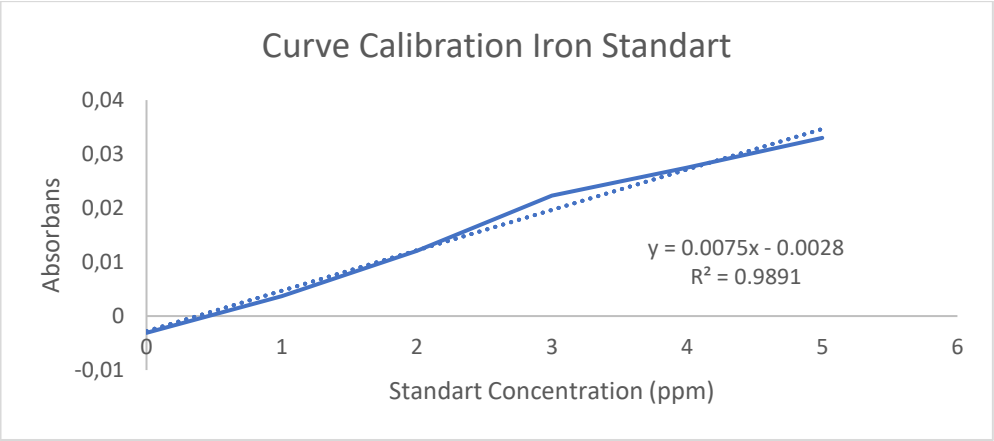

Figure 1. Curve calibration Iron standard

Table 2. Results of analysis of Fe(III) using AAS

Sample Solution	Repetition	[Sample] (ppm)	Absorbance	Absorbance Mean	Fe content (mg/capsule)	Content Mean	Deviation Standard
0,5 ml	1	1.51	0.0085	0.00967	32,53	35,89	3,62
	2	1.84	0.011		39,73		
	3	1.64	0.0095		35,41		
1 ml	1	2.68	0.0173	0.0156	28,93	26,53	2,20
	2	2.41	0.0153		26,05		
	3	2.28	0.0143		24,61		

Equation

In determining the standard curve using AAS will get a linear regression equation, with the equation

$$y = ax + b \tag{1}$$

By utilizing this equation, a new equation will be obtained to determine the concentration of the sample solution

$$x = \frac{y - b}{a} \tag{2}$$

$$Concentration = \frac{Absorbance - b}{a} \tag{3}$$

With

$$y = 0.0075x - 0.0028$$

Then

$$Concentration = \frac{Absorbance + 0.0028}{0.0075} \tag{4}$$

Obtained the value of content Fe in Table 2

in determining the sensitivity and selectivity we can test with the t test, which is compared between the actual value with the value of the analysis results with the equation

$$t_{hitung} = \frac{\bar{x} - \mu_0}{s/\sqrt{n}} \quad (5)$$

With

$$Ho; \mu = 38\ mg/capsule$$
$$\text{H1}; \mu \neq 38\ mg/capsule$$

95% significance level or α= 0,05, then

$$df = n - 1 = 3 - 1 = 2$$
$$t_{tabel} = 4.30$$

for sample 0.5 mL

$$t_{hitung} = \frac{\bar{x} - \mu_0}{s/\sqrt{n}} = \frac{35.89 - 38}{3.62/\sqrt{3}} = 1.005$$

for sample 1 mL

$$t_{hitung} = \frac{\bar{x} - \mu_0}{s/\sqrt{n}} = \frac{26.53 - 38}{3.62/\sqrt{3}} = 9.03$$

for sample 0.5 ml

$$t_{hitung} < t_{tabel}$$

Then it fails to reject H_0
As for the 1 mL sample

$$t_{hitung} > t_{tabel}$$

Then Reject H_0

The solution made with a sample volume of 0.5 mL (low concentration) has a high sensitivity, this is proven by the t test. on the t test, it was found that Ho was accepted, this was due to the value, $t_{count} < t_{table}$. whereas a solution with a sample volume of 1mL (higher concentration) has a lower sensitivity, as evidenced by the results of the t test. In the t test the value of Ho is rejected which results in H1 being accepted, because the value of $t_{count} < t_{table}$. It can be concluded that for samples with small concentrations, the AAS instrument can measure sensitively whereas when using high concentrations, the instrument is less sensitive in its measurement which results in the actual mass of iron in the multivitamin tablet not being the same as the mass tested.

RESULTS AND DISCUSSION

This experiment was conducted to determine the levels of iron (Fe) contained in blood booster tablets. Measurement of iron content was carried out using Atomic Absorption Spectroscopy (AAS). First of all, standard solutions were made with concentrations of 0, 1, 2, 3, 4, and 5 ppm. The standard series used are different in order to make a linear regression equation so that the concentration of the sample can be determined. The sample solution is prepared by dissolving Multivitamin Tablet with HNO3. The aim is that the multivitamin tablet is completely dissolved because this multivitamin

tablet is difficult to dissolve in water. Dissolving the sample using HNO3 also aims to prevent the formation of Fe(OH)3 precipitates. In water, iron ions can experience hydrolysis and form Fe(OH)3 precipitates which can reduce the accuracy of the measurement. Heating function in order to accelerate the extraction of iron and elements contained in the sample.

The working principle of atomic absorption spectrophotometry is based on the evaporation of the sample solution which will initially be nebulized to form a spray, then it will be desolvated to form a dry aerosol until it is evaporated and converted into free atoms. The flame then burns the atoms so that the atoms can absorb the light emitted from the cathode lamp containing the element to be determined. The amount of radiation absorption is then measured at certain wavelengths according to the type of metal (Wilberforce et al., 2016). The reduction in the intensity of a given radiation is proportional to the number of atoms of the fundamental energy level in absorbing the radiation energy. By measuring the intensity of transmitted radiation or measuring the intensity of absorbed radiation, the concentration of the elements inside is determined by stating the relationship between the concentration of the solution and its absorbance. This is in accordance with the Lambert-Beer law which states that the amount of energy absorbed will be proportional to the concentration (Bhanvase & Barai, 2021).

Determination of Fe3+ levels in previous studies has been carried out by the UV-Vis spectrophotometry method. From these results, the average iron level in blood booster supplements was 69.626 mg/capsule. This shows that there is a quite clear difference when compared to the results obtained by atomic absorption spectrophotometry method. In addition, differences in sensitivity levels can also be another cause. AAS has a better level of sensitivity compared to UV-Vis spectrophotometry(Qassim et al., 2011). Judging from the packaging of the blood booster tablets tested, it was written that the Fe level was 38 mg/capsule. This value is not much different from the results of measuring iron levels using AAS to obtain iron levels at a volume of 0.5 mL obtained around 35.891 mg/capsule while for a volume of 1 mL around 26.53 mg/capsule. Quite different values were obtained when the measurement of levels was carried out using the UV-Vis spectrophotometry method(Qassim et al., 2011). With this method, the iron levels that were measured were quite different from the iron levels listed on the packaging. The success of using AAS depends on the excitation process and how to get the right resonance line(Fernández et al., 2019).

Based on the observations, Table 1 shows that the greater the standard concentration of Fe, the greater the absorbance level. Figure 1 represents the Fe Ion standard curve so that the regression equation $y = 0.0075x - 0.0028$ is obtained; R2 = 0.9891. This shows that the resulting linearity data is acceptable because it is > 95% (Nashukha et al., 2014). The absorbance of the sample solution obtained is then substituted into the standard curve regression equation with x as concentration and y as absorbance. It was found that the average Fe/capsule level in 3 replicates for each type of sample was 35.891 mg/capsule with a standard deviation of 3.62259 which is shown in table 2 of the experimental results. Based on the literature, it was found that the Fe level in the multivitamin capsule was 30.98 mg/capsule using the same method, namely AAS. This shows that there is no significant difference between the experimental results and the literature.

In determining the selectivity of a tool, a 2-way hypothesis test can be carried out with different concentrations. When multivitamin tablets is made into a solution with the same concentration but different volumes taken. In this study the volumes taken and tested were 0.5 mL and 1 mL volumes. The absorbance of the sample was then measured using AAS and using the regression equation to obtain the actual concentration and then converted to mass, now this mass is compared to the weight of iron in the Multivitamin Tablets, where the Multivitamin Tablets has a mass of iron of around 38 mg/capsule. A 2-way hypothesis test is obtained. The solution made with a sample volume of 0.5 mL (low concentration) has a high sensitivity, this is proven by the t test. on the t test, it was found that Ho was accepted, this was due to the value, tcount <ttable . whereas a solution with a sample volume of 1mL (higher concentration) has a lower sensitivity, as evidenced by the results of the t test. In the t test the value of Ho is rejected which results in H1 being accepted, because the value of tcount <ttable . It can be concluded that for samples with small concentrations, the AAS instrument can measure sensitively whereas when using high concentrations, the instrument is less sensitive in its measurement which results in the actual mass of iron in the multivitamin tablet not being the same as the mass tested

CONCLUSION

Iron levels in a volume of 0.5 mL were found to be around 35.891 mg/capsule while for a volume of 1 mL it was around 26.53 mg/capsule. Determination of levels in multivitamin tablets can be done by the absorption atomic spectroscopy(AAS) method. Based on the experimental results, the atomic absorption spectrophotometry method has a higher level of accuracy and activity compared to UV-Vis spectrophotometry. Iron levels in samples using AAS can be found by measuring the intensity of the radiation that decreases after being transmitted. The reduction in radiation intensity is proportional to the concentration of the sample element in the sample being measured. By testing the t test, it was found that the AAS instrument was more sensitive to low concentrations.

For further research, it can be done by using other methods and other instruments such as using UV-Vis spectroscopy using chelatometry. Another approach, addition of complex ions to clearly determine the level of iron contained in multivitamin tablets can be done.

AUTHOR CONTRIBUTIONS

Each author of this article played an important role in the process of method conceptualization, simulation, and article writing.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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