



DEVELOPMENT OF TEST KIT SOLUTIONS TO DETERMINE IRON CONTENT IN NATURAL WATER SOURCES

Tran Le Ba¹

¹An Giang University, VNU-HCM

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ABSTRACT

In this study, a solution-test kit was developed to determine a variety of iron contents and give quick results on site. Three color tables representing different iron contents were studied: color table 1 (0–1 mg/L), color table 2 (0–10 mg/L), and color table 3 (0–100 mg/L), with each color table corresponding to a specific test kit. The results showed a high linearity between color intensity and iron content from the 3 color tables, with the results always greater than 0.9. In addition, this test kit can also determine the content of different types of iron (Fe^{2+} , Fe^{3+} and total iron) in water sources. The comparison results of the iron content prepared in the laboratory (S1, S2, and S3 samples) analyzed using the test kit (through the color intensity created from $Fe(SCN)_3$ and the analysis method TCVN 6177: 1996 (ISO 6332: 1988 (E) showed that the deviation was less than 5%. Besides, the iron-containing water samples (SN1, SN2, and SN3) from nature also gave similar deviation results to those of samples created in the laboratory. Moreover, with the color differences in the chart, the test kit can quickly determine the iron content, similar to the pH determination method using a color chart. The test kit has demonstrated its high applicability by determining the iron content over a wide range (from 0 to 100 mg/L) to assess water quality for agricultural and aquaculture activities.

1. INTRODUCTION

Iron is a metal commonly found in water. It is the fourth most common element on Earth and the second most abundant in the Earth's crust. Iron is present in water due to geological processes as well as human activities and production. Iron exists in both surface water and groundwater (from rocks and minerals containing iron) (N. Khatri et al., 2017). In Vietnam, depending on different regions, the iron content in surface water can vary from 0 mg/L to more than 40 mg/L (Trung Thanh Nguyen et al., 2021) and iron contents in

groundwater can exceed 20 mg/L. This variation in iron content has directly affected daily activities (through bathing, washing, eating, etc.) as well as agricultural, aquaculture, and livestock production activities when the iron content in water exceeds permissible limits. According to the QCVN 01-1:2018/BYT standard of the Vietnamese Ministry of Health which sets the national technical regulation on domestic water quality, the allowable iron content in water is 0.3 mg/L. For groundwater, the allowable iron content is 5 mg/L (according to QCVN 09:2023/BTNMT, set by the Ministry

of Natural Resources and Environment of Vietnam). In particular, the iron content in drinking water is often higher than the allowable level, which can cause gastrointestinal diseases. Additionally, iron ions in water can promote the growth of iron bacteria. These bacteria produce reddish-brown slime that can clog water systems. Furthermore, iron compounds create reddish-brown stains on clothes and household items such as dishes, glassware and sinks, etc. (M. L. McFarland and M. C. Dozier, 2024). Therefore, it is necessary to have a method to determine the iron content in water to implement appropriate management and treatment measures for iron-contaminated natural water sources. In previous studies, test kits have been developed to determine the iron content using both solution and paper (Le Ba Tran et al., 2023; S. Annem, 2017 & P. Patil et al., 2012). However, the test kits studied typically determine iron content up to only 10 mg/L and are costly. They often use a visual colorimetric method that utilizes o-Phenanthroline which combines with Fe^{2+} to form an orange-red complex. Test kit solutions are commonly used in field analysis due to their high efficiency and stability.

In this study, we developed a solution-based test kit to determine the iron content in water. The test kit includes 3 separate color tables representing concentration of 0-1 mg/L; 0-10 mg/L and 0-100 mg/L. The color of the solution results from the interaction between Fe^{3+} ions and SCN^- ions, forming a red compound ($FeSCN_3$). The test kit can simultaneously determine the content of Fe^{3+} , Fe^{2+} and total iron in the same solution. This study is necessary to diversify the methods of rapid iron content testing in the field, helping to shorten the time and reduce the cost of iron analysis.

2. MATERIALS AND METHODS

2.1 Chemicals and water sources

Chemicals used for research include: KSCN, $Fe(SO_4)_3$, H_2O_2 , H_2SO_4 , HNO_3 , HCl, CH_3COONH_4 , $NH_2OH.HCl$, $C_{12}H_9ClN_2.H_2O$, and $K_2S_2O_8$, which were bought from Merck.

Surface water containing iron was collected in Vinh Phuoc commune, Tri Ton district, An Giang province, Vietnam. The locations sampled at 3 specific Google coordinates were as follows: SN1 (10.442405, 104.887461), SN2 (10.440929, 104.888577), and SN3 (10.459249 104.877001) and 3 water samples containing iron were synthesized at the laboratory of An Giang University. Natural water samples containing iron were collected following the TCVN 6663-6:2008 (ISO 5667-6: 2005) standards on water quality sampling - Part 6: Guidelines for sampling river and stream water. Then, the water samples were acidified and stored at 4°C before analysis.

2.2 Analytical method for iron content in water samples

Analysis according to applicable standards

The water samples containing iron were analyzed according to TCVN 6177:1996 (ISO 6332:1988) using 1,10-phenanthroline reagent. However, before determining the iron content, the water samples were filtered through filter paper, the pH was adjusted, and the subsequent steps were performed according to the analysis instructions.

Production of color indicator solution

Color indicator solution for iron content (0-1 mg/L) - Solution A

The color indicator solution consists of 3 components: SL_{1a} , SL_{1b} and SL_{1c} . SL_{1a} was prepared using 9,57 g KSCN dissolved in 25 mL of deionized water. SL_{1b} was made by mixing 10 mL of 30% H_2O_2 with 240 mL of deionized water, and while SL_{1c} was created by combining 10 mL of 65%-68% HNO_3 with 40 mL of deionized water.

Color indicator solution for iron content (0-10 mg/L) - Solution B

The color indicator solution includes 3 components: SL_{2a}, SL_{1b} and SL_{2b}. SL_{2a} was prepared using 9.57mg of KSCN dissolved in 100 mL of deionized water, while SL_{2b} was made by mixing 20 mL of 65-68% HNO₃, with 180 mL of deionized water.

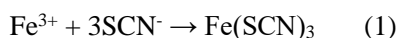
Color indicator solution for iron content (0-100 mg/L) - Solution C

The color indicator solution includes 2 components: SL_{3a} and SL_{1b}. SL_{3a} was prepared using 4.785 g of KSCN dissolved in 180 mL of deionized water.

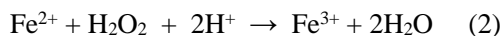
The color indicator solution was kept stable at room temperature, while the SL_{1b} solution was stored in low light conditions.

2.3 Mechanism for color formation in iron determination

The method of determining color is based on the chemical reaction between Fe³⁺ ions and SCN⁻ ions, which produces Fe(SCN)₃, a compound that exhibits a prominent blood red color, as shown in the following equation (Pa Ho Hsu, 1967; K. Ozutsumi et al., 1992 & K. S. Patel et al., 2001):



In addition, the Fe²⁺ ion, when combined with SCN⁻ ion, will form the Fe(SCN)₂ compound; this compound has no characteristic color. Therefore, it is necessary to convert Fe²⁺ ions into Fe³⁺ ions using H₂O₂ solution, as shown in the following specific equation:



2.4 Method for determining color intensity by imaging in iron analysis

Images from color charts and images from experimental samples were taken with a digital camera (Cannon Inc., Tokyo, Japan). The colors

in the image were represented through the mixture of three primary colors: red, green, and blue (RGB). The color intensity value (G) of the sample was quantified based on the RGB values. The values of these 3 colors on the sample image were obtained through the use of free color matching software (Pixie, Version: 4.1, Nattyware) (Nattyware, 2022). Next, these values will be converted to Lab color space by a free online software program (The Ultimate Color Translator, Nix Sensor Ltd, Canada), resulting in lightness (L), color opponent green-red (a), and color opponent blue-yellow (b) values (Le Ba Tran et al., 2023 & Nix Sensor Ltd, 2022). The color difference (ΔE) expressed by the formula (Le Ba Tran et al., 2023; R. S. Hunter, 1958 & M. Sekine et al., 2018):

$$\Delta E = \sqrt{(\Delta L^2 + \Delta a^2 + \Delta b^2)} \quad (3)$$

Where ΔL , Δa , and Δb are the difference L, a and b values between iron-containing water samples and iron-free water samples. This allows for comparing the color of the treated water sample with the color indicator solution to the created color charts to find the iron content, or to determine the ΔE value for more accurate iron content estimation.

2.5 Statistical analysis

All parametric analyses and statistical values in the study were performed using Microsoft Office 2016 software, and Origin 2018 software. Due to the small sample size, non-parametric tests were applied. The iron content in water samples was determined using a color indicator solution with quantitative image analysis—and TCVN 6177: 1996 (ISO 6332: 1988 E) method, conducted at An Giang University. All analyzed samples were repeated 3 times.

3. RESULTS AND DISCUSSION

3.1 Method for determining iron content by color indicator solution

Method for determining iron content (0-1 mg/l)

The method of coloring iron-containing water samples was performed as follows:

For the process of determining Fe^{3+} : 5 mL of the iron-containing water sample was combined with 0.25 mL of PL_{1c} . Then, 1 mL of PL_{1a} was added, and the solution was shaken well. The sample was left undisturbed for 30 seconds, and the color of the solution was determined.

For the process of determining total iron: 5 mL of the iron-containing water sample was combined with 0.25 mL of PL_{1b} . Then, 0.25 mL of PL_{1c} and 1 mL PL_{1a} were added. The solution well, and the sample was kept stable for 30 seconds. Afterward, the color of the solution was determined.

The Fe^{2+} content was determined by subtracting the Fe^{3+} content from the total iron content in the iron-containing water sample.

Method for determining iron content (0-10 mg/l)

The method of coloring iron-containing water samples was performed as follows:

To determine Fe^{3+} : mix 5 mL of the iron-containing water sample with 0.25 mL of PL_{2b} , followed by 0.25 mL of PL_{2a} . Shake well and let the solution sit for 30 seconds before checking the color.

For total iron: mix 5 mL of the water sample with 0.25 mL of PL_{1b} , 0.25 mL of PL_{2b} , and 0.25 mL of PL_{2a} . Shake well, wait 30 seconds, then check the color.

Fe^{2+} content was determined by subtracting the Fe^{3+} content from the total iron content.

Method for determining iron content (0-100 mg/l)

To determine Fe^{3+} and total iron, mix 5 mL of the iron-containing water sample with 0.25 mL of PL_{3a} or 0.25 mL of PL_{1b} and 0.25 mL of PL_{3a} respectively, let it sit for 30 seconds, and then check the color, while Fe^{2+} is calculated by subtracting the Fe^{3+} content from the total iron content.

3.2 Create color charts for color indicator solutions

The purpose of this study was to create a color indicator solution to determine the iron content in water. To achieve this, it was necessary to develop a color chart of color indicator solutions corresponding to varying concentrations of iron (III) in the water environment. The color chart was built with three sets corresponding to different ranges of iron content: 0-1 mg/L, with a step of 0.1 mg/L; 0-10 mg/L, with a step of 1 mg/L; and 0-100 mg/L, with a step of 10 mg/L. The results of creating these 3 color charts using the color indicator solution were shown in Table 1. In general, all three color charts indicate that after applying the color indicator solution, the iron-containing solution changes from colorless (indicating no color change compared to the original iron-free solution) to red, corresponding to an increase in iron (III) concentration from low to high. This demonstrates that the study successfully developed three color charts for different concentration of iron (III). However, to enhance the accuracy of quantifying iron (III) content in water samples, it is crucial to employ image analysis methods utilizing cameras or mobile phones combined with image analysis software to detect chromatic aberration, which is extremely importance in practice.

Table 1. Paper color chart corresponding to iron (III) content from 0 to 100 mg/L

No.	Fe ³⁺ content*	Color palette	Fe ³⁺ content*	Color palette	Fe ³⁺ content*	Color palette
1	0 mg/L		0 mg/L		0 mg/L	
2	10 mg/L		1 mg/L		0.1 mg/L	
3	20 mg/L		2 mg/L		0.2 mg/L	
4	30 mg/L		3 mg/L		0.3 mg/L	
5	40 mg/L		4 mg/L		0.4 mg/L	
6	50 mg/L		5 mg/L		0.5 mg/L	
7	60 mg/L		6 mg/L		0.6 mg/L	
8	70 mg/L		7 mg/L		0.7 mg/L	
9	80 mg/L		8 mg/L		0.8 mg/L	
10	90 mg/L		9 mg/L		0.9 mg/L	
11	100 mg/L		10 mg/L		1 mg/L	

Note: *: theoretically prepared content using Standard iron solution (Germany).

3.3 Construct a linear curve between iron (III) content and color intensity of the solution

In the imaging quantification method, constructing a linear calibration curve between color intensity and iron (III) content is a crucial step for determining the iron (III) concentration in water. By measuring the color intensity of iron-containing water samples, it the iron

content can be deduced from the linear equations derived from the calibration curve of three color tables.

Three standard color tables were created, each corresponding to different ranges of iron (III) content: Standard Color Table 1 for iron (III) concentration from 0-1 mg/L (with a increments of 0.1 mg/L), corresponding to test kit solution A for measuring iron content below 1 mg/L;

Standard Color Table 2 for iron (III) concentration from 0-10 mg/L (with increments of 1 mg/L), corresponding to test kit solution B for measuring iron content below 10 mg/L and Standard Color Table 3 for iron (III) concentration from 0-100 mg/L (with increments of 10 mg/L) corresponding to test kit solution C for measuring iron content below 100 mg/L.

By using Excel 2016 software, linear equations were established to relate color intensity to iron content in water (Figure 1). Specifically the equations included: for iron content from 0-1 mg/L, the linear equation $\Delta E = 19.048 x - 0.0791$ ($R^2 = 0.9089$); for iron content from 0-10

mg/L the linear equation is $\Delta E = 6.3673 x - 1.3829$ ($R^2 = 0.9907$) and for iron content from 0-100 mg/L the equation is $\Delta E = 1.0095 x - 0.869$; ($R^2 = 0.9923$), where x represents iron content (mg/L) and ΔE denotes the color intensity of the iron-containing water sample. The results from these three equations demonstrate that the image analysis method can to effectively determine iron content in the range of 0 - 100 mg/L. In addition, the correlation coefficients for all the models exceed 0.9, indicating a strong linear relationship between iron content and color intensity, and suggesting that the results from the image analysis process were are highly reliable.

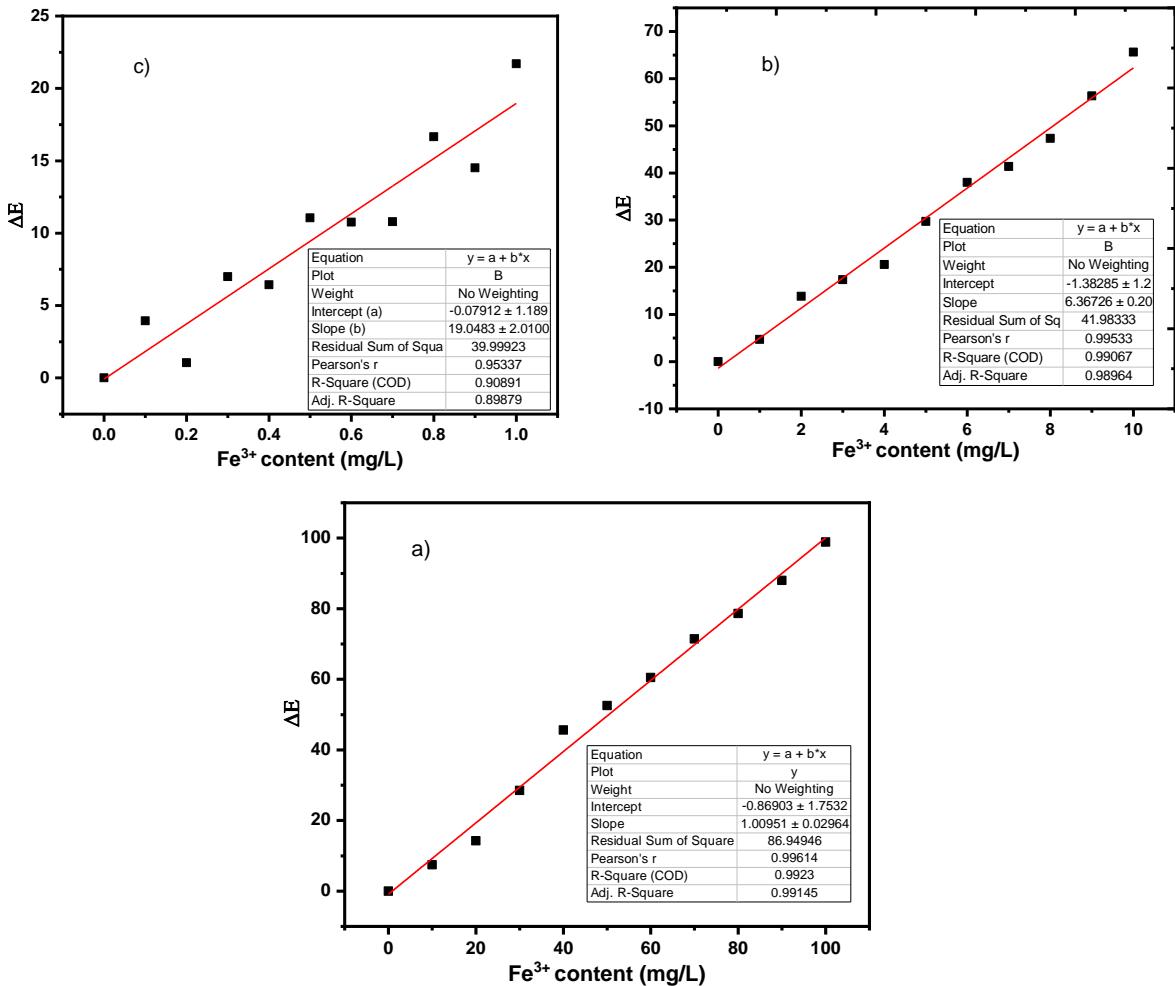


Figure 1. Linear equations of image analysis for iron quantification. (a: 0 to 100 mg/L, b: 0 to 10 mg/L, and c: 0 to 1 mg/L)

3.4 Determination of iron content in natural water by image analysis method

This study was divided into 2 parts: Part 1 involved determining the iron content of 3 water samples containing Fe³⁺ (S1, S2 and S3) created in the laboratory, while Part 2 focused on determining the iron content in 3 natural water samples (SN1, SN2 and SN3).

For the laboratory-created water samples containing Fe³⁺, the results from the color indicator solution analysis were relatively consistent with those obtained using 1,10-phenanthroline reagent.

Specifically, sample S1 showed an Fe³⁺ content of 0.419 mg/L with a deviation of about 5%; sample S2 had an Fe³⁺ content of 4.968 mg/L with a deviation of about 4%;

Specifically, sample S1 showed an Fe³⁺ content of 0.419 mg/L with a deviation of about 5%, sample S2 had an Fe³⁺ content of 4.968 mg/L with a deviation of about 4%. And sample S3 exhibited a content of 26.328 mg/L with a deviation of approximately 4%. The laboratory analysis of iron-containing water samples yielded highly reliable results, with a deviations consistently less than 5%. Furthermore, the analysis of Fe²⁺ content in the laboratory created water samples using a the color indicator solution also produced very low error results (less than 5%) compared to the method using the 1,10-phenanthroline reagent.

When analyzing natural iron alum water samples with the color indicator solution method, the Fe³⁺ content in samples SN1, SN2, and SN3 was found to be 6.652 mg/L, 5.328 mg/L and 7.665 mg/L, respectively. These analytical results showed a relatively small deviation (less than 5%) when compared to the results obtained using the 1,10-phenanthroline reagent.

4. CONCLUSIONS

The study successfully produced three sets of color indicator solutions for determining the presence and concentration of iron in naturally contaminated water. Analysis of Fe³⁺ content in both laboratory created samples and actual Fe³⁺-contaminated water samples yielded ~~ve~~ results with an error margin of less than 5% when compared to the 1,10-phenanthroline reagent method. The method is beneficial-for scientists, water resource managers and farmers dealing with iron-contaminated water sources, enabling them to effectively manage, evaluate, and classify these sources for various applications.

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