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Development of facile field-test paper for rapid determination of iron contamination in natural water of acid sulfate soils

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ABSTRACT

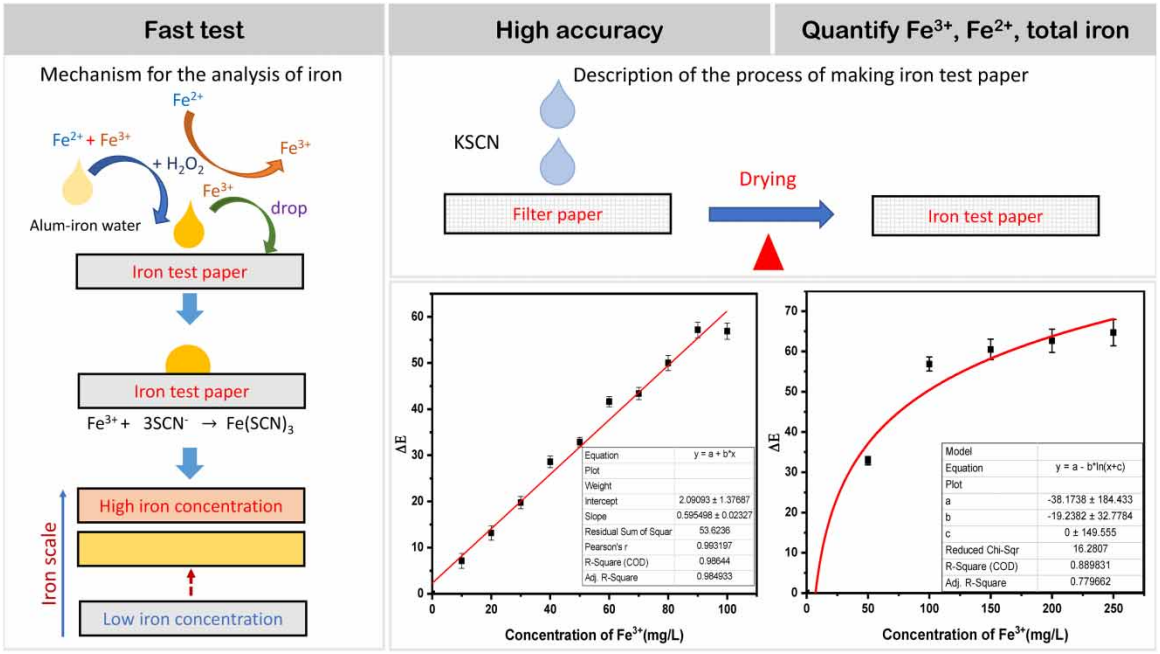
The iron concentration in natural water of acid sulfate soils is usually determined by complicated methods of SMEWW 3111B:2017 or ISO 6332: 1988 E in the laboratory for acidified water samples at pH ~2–3. Therefore, the rapid field determination of iron content in natural water is a very useful method because it limits the error and reduces the experimental time for analysis. In this study, we proposed, developed, and tested a rapid colorimetric analysis method based on the chemical reaction of ferric and anion thiocyanate (SCN^-) ions for an iron concentration range of 0–100 mg/L with a linear equation of $\Delta E = 0.5955x + 2.0909$ and high correlation coefficient (R^2) of 0.9864. The results from 10 water samples by this method were very close to those by flame absorption spectroscopy with a difference of less than 5%. Besides the biggest advantage of rapid site testing, this method also gives quite high accuracy compared to the molecular absorption method of ISO 6332:1988 E. In addition, this new method can quantify Fe^{3+}, Fe^{2+}, and total iron in water, suggesting a very useful and potential method for determining the iron content of natural water from acid sulfate soils.

Key words: acid sulfate soil, color chart range, colorimetric analysis, iron analysis, test kit

HIGHLIGHTS

- A color chart was developed to determine iron in water using indicator paper.
- A linearity of the iron content was obtained with a concentration below 100 mg/L.
- The method gives relatively fast and accurate results of Fe^{2+}, Fe^{3+}, and total iron.
- The tested results show high accuracy and sensitivity in natural iron alum water.

GRAPHICAL ABSTRACT



1. INTRODUCTION

The natural water of acid sulfate soils (named natural iron alum water) is usually of high iron ion content and low pH value. This water has a great impact on human health, water environment, and local agricultural production activities (Vuori 1995; Walker & Walker 2000; Moore Heslin *et al.* 2021), which is generally distributed in Southeast Asian countries and Vietnam. The water composition of natural iron alum water greatly depends on the stratigraphic structure of the soil (Nguyen *et al.* 2021), which can be divided into two types of (1) iron alum water with a high content of iron and (2) aluminum alum water with high content of aluminum ions. However, these two types of natural iron alum water can be distinguished by observation. Aluminum alum water has only a bluish-white color, while iron alum water is usually in pink or red color, depending on its iron content. Natural iron alum water can be divided into three types based on its iron content, including type 1 (30–120 mg/L, yellow color, pH 2.5–3.0), type 2 (25–70 mg/L, opaque yellow color, many impurities, pH 2.5–3.5), and type 3 (2–10 mg/L, blue color, pH 2.5–2.8).

When performing the determination of iron content in natural iron alum water, absorption spectrophotometric methods with reagents of 1,10-phenanthroline according to TCVN 6177:1996 (ISO 6332:1988 E) were commonly used, and natural iron alum water samples before being brought to the laboratory must be fixed with a certain amount of acid to avoid precipitation of iron and other metal ions (I. O. f. Standardization 1988). In other words, the determination of iron ion content in natural iron alum water has not been done in the field. This limitation may be that there has not been a suitable analytical method and/or instrument/equipment to perform the in situ determination of iron content in natural iron alum water in the field. In summary, the studies conducted to search for analytical methods and design compatible tools/devices to determine iron content in natural iron alum water were still a great challenge for scientists to reduce analysis time and cost and improve the accuracy in the process of determining iron alum content in natural iron alum water in the field.

In this study, we developed and applied a colorimetric method based on the reaction between iron ions and thiocyanate ions impregnated on filter paper combined with an image analysis method for the quantitative determination of natural iron alum water with high accuracy, which was compared to the results from the conventional method of TCVN 6177: 1996 (ISO 6332: 1988 E). This method can be performed in the field to meet the requirements of many scientists in many fields who are conducting research related to the iron alum water environment. In general, this method would be an appropriate and convenient method to determine the iron content in natural iron alum water in the field.

2. MATERIALS AND METHODS

2.1. Chemicals and water sources

The filter paper used in this study was 102-Whatman. Chemicals such as potassium thiocyanate (KSCN), iron(III) sulfate ($Fe_2(SO_4)_3$), hydrogen peroxide (H_2O_2), acid sulfuric (H_2SO_4), acid nitric (HNO_3), hydrochloride (HCl), ammonium acetate (CH_3COONH_4), hydroxylamine hydrochloride (NH2OH.HCl), 1,10-phenanthroline (phen) ($C_{12}H_9ClN_2.H_2O$), and potassium persulfate ($K_2S_2O_8$) used in this study were bought from Merck.

Natural iron alum water was collected in Vinh Phuoc commune, Tri Ton district, An Giang province, Vietnam. The natural iron alum water samples were taken at 10 locations: S1 (10.42300042676598, 104.84797608994386), S2 (10.428206, 104.840027), S3 (10.455291, 104.866944), S4 (10.447852, 104.861008), S5 (10.440103, 104.855177), S6 (10.433025, 104.849871), S7 (10.425741, 104.844251), S8 (10.419541, 104.839523), S9 (10.412308, 104.834375), and S10 (10.406676, 104.829647). These water samples were taken according to TCVN 6663-6:2008 (ISO 5667-6: 2005) – Water quality – Sampling – Part 6: Guidelines for sampling river and stream water. Then, the water sample was acidified and stored at 4 °C in the laboratory. The composition of natural iron alum water was determined by the flame emission spectroscopy method (according to TCVN 7207-5:2002) with composition (heavy metal) as shown in Table 1. The composition of natural iron alum water in this study has similar results to the previously published results of our group (Nguyen *et al.* 2021).

Table 1 | Contents of some typical metal ions present in natural iron alum water at Vinh Phuoc commune, Tri Ton district, An Giang province, Vietnam

Elemental	Fe	Al	Ca	Mg	Si
Concentration (mg/L)	41.45	5.67	184.1	452.15	5.07

2.2. Analytical method for Fe(III) content in water samples

The content of iron(III) in water samples (water samples containing only iron(III) ions and natural iron alum water) was performed by molecular absorption spectroscopy (colorimetric method) according to TCVN 6177:1996 (ISO 6332:1988) E) with a reagent of 1,10-phenanthroline. However, before performing analysis to determine the iron(III) content, water samples were filtered through filter paper, adjusted the pH value, and analyzed according to the instructions of the analytical method.

2.3. Production of iron indicator paper

Iron indicator paper was made from ordinary filter paper (102 – Whatman – Ø150 mm). The filter paper was washed with deionized distilled water three times and allowed to dry naturally. Then, the paper was evenly impregnated with the KSCN solution (2 M) by immersing in the solution and dried at 105 °C for 4 h. This process was repeated three times (Huygen 1963; Chardon *et al.* 1996; Qin *et al.* 2019). When the paper sample met the requirements for KSCN impregnating, it was cut into small pieces (size of 1 × 4 cm) and stored at room temperature in a desiccator to serve the next experiments.

2.4. Method of analyzing iron content by the color table

In this study, the method of determining iron content by the colorimetric method is based on the reaction between Fe^{3+} ion and SCN^- ion as described in the following equation, and the product of this reaction is blood red (Hsu 1967; Ozutsumi *et al.* 1992; Patel *et al.* 2001; Filgueiras & Borges 2022).

$$Fe^{3+} + 3SCN^- \rightarrow Fe(SCN)_3 \quad (1)$$

Besides, Fe^{2+} ion also reacts with SCN^- ion. However, the product of this reaction is colorless.

$$Fe^{2+} + 2SCN^- \rightarrow Fe(SCN)_2 \quad (2)$$

2.5. Quantification of iron using image analysis

The image of the test paper and color palette of the iron test kit was taken with a digital camera in the field (Cannon Inc., Tokyo, Japan). The colors on the test paper samples are represented by combining the three primary colors of red, green, and blue (RGB). The values of color intensity per test paper and standard colors were quantified in RGB values using free software (Pixie, Version: 4.1, Nattyware) (Nattyware 2022). The RGB values of the test paper samples found with Pixie software are converted to Lab color space, with the names of lightness (L), color opponent green-red (a), and color opponent blue-yellow (b) values, by a free online software program (the ultimate color translator, Nix Sensor Ltd, Canada) (Nix Sensor Ltd 2022). The color difference, ΔE, was calculated based on the values of Lab color space (where the color value of the iron sample with a content of 0 mg/L was used as a control with the other values) according to the following equation (Hunter 1958; Sekine *et al.* 2018):

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

where ΔL, Δa, and Δb are the difference values of L, a, and b between the iron alum sample and the control sample, respectively. The ΔE value is determined on the test paper for the water sample at the sampling site and the standard color on the color chart range. The iron(III) concentration was then determined using a standard curve that was determined in the laboratory. Besides, this method is also checked for recovery through the following equation:

$$R(\%) = \frac{C_s - C}{S} \tag{4}$$

where C_s, C, and S are the values of the concentration of the added standard, the concentration of the matrix (field sample), and the concentration of the standard when added, respectively (APHA, AWWA, WEF 2017).

2.6. Statistical analysis

All parametric analyses and statistical values in the study were performed using Microsoft 365 software. Because of the small sample size, nonparametric tests were used in the calculation. Spearman's rank-order correlation coefficient was used correlation when determining iron concentration by our field test kit with the quantitative image analysis method and the TCVN 6177: 1996 (ISO 6332: 1988 E) method at An Giang University. All analyzed samples were repeated three times. The drawings were made using Origin 2018 software from OriginLab Corporation.

3. RESULTS AND DISCUSSION

3.1. Standard curve using quantitative image analysis method

Color indicator paper according to iron(III) ion concentration was made by impregnating the filter paper with KSCN by a simple process as shown in the experimental section (Shapiro 1969). Visually, it was shown that the filter paper before and after impregnating KSCN had no difference in color (white).

3.2. Building a color palette for iron indicator paper

As mentioned earlier, the main content of this research is to fabricate color indicator paper to determine iron ion content in an aqueous medium. Therefore, it is necessary to develop a color chart of the indicator paper corresponding to the iron(III) concentration in the water. The color chart of the paper was constructed corresponding to the iron(III) concentration from 0 to 100 mg/L, with a step of 10 mg/L. The results of constructing the color palette of the paper are presented in Table 2. From this result, it was shown that the color of the paper changed color from white (no change in color from the original one) to blood red, corresponding to the iron(III) concentration changing from 0 to 100 mg/L. In addition, the color of the palette had a marked color difference between the two different iron(III) concentrations. This color was the color of $Fe(SCN)_3$, which is the product of the reaction between Fe^{3+} and SCN^- ions, and the color was very stable under atmospheric conditions. From this result, it can be said that a color table corresponding to iron(III) concentration has been successfully constructed. However, to increase the accuracy in quantifying iron(III) content in water samples, the application of imaging methods combined with modern and portable devices to distinguish chromatic aberration is essential in the field.

Table 2 | Paper color chart corresponding to iron(III) concentration from 0 to 100 mg/L

No.	Fe^{3+} concentration (mg/L)[a]	Color palette	Analyzed concentration[b] (mgFe^{3+}/L)
1	0		0
2	10		9.8 ± 0.24
3	20		18.6 ± 0.36
4	30		27.5 ± 0.35
5	40		38.6 ± 0.44
6	50		48.9 ± 0.48
7	60		57.4 ± 0.56
8	70		68.3 ± 0.78
9	80		77.6 ± 0.64
10	90		87.2 ± 0.92
11	100		98.9 ± 0.88

Note: [a]Theoretically prepared concentration using pure chemicals.
[b]Analytical results at VILAS laboratory according to TCVN 6177:1996 (ISO 6332:1988 E).

3.3. Construction of standard curve of iron(III) quantification method by image analysis

The construction of a standard curve by the imaging method is a very important operation to quantify the iron(III) content in the water environment. For this quantitative method of image analysis, the color difference between the sample and the control color histogram in the laboratory is compared to determine the concentration of iron in the water sample. Standard iron(III) concentration ranging from 0 to 100 mg/L (with a step of 10 mg/L) was prepared to generate a standard table for color indicator paper according to iron(III) ion concentration used in image quantification.

The results from the equation calculated by Excel software showed a linear standard curve ($\Delta E = 0.5955x + 2.0909$; $R^2 = 0.9864$) up to a concentration of 100 mg/L (Figure 1(a)). When the iron content was in the range of 0–250 mg/L, the standard curve becomes the curve and the linear equation of the experiment gives $R^2 = 0.8898$ (Figure 1(b)), where x is the iron concentration (mg/L) and ΔE is the color difference. The results from these two equations showed that this imaging method is able to determine the iron(III) content in water in the concentration of 0–100 mg/L.

3.4. Determination of iron(III) content in natural iron alum water by image analysis method

In natural iron alum water, iron is considered the main metal that affects the properties of natural iron alum water. When the iron content changes, the quality of natural iron alum water also changes in the same direction.

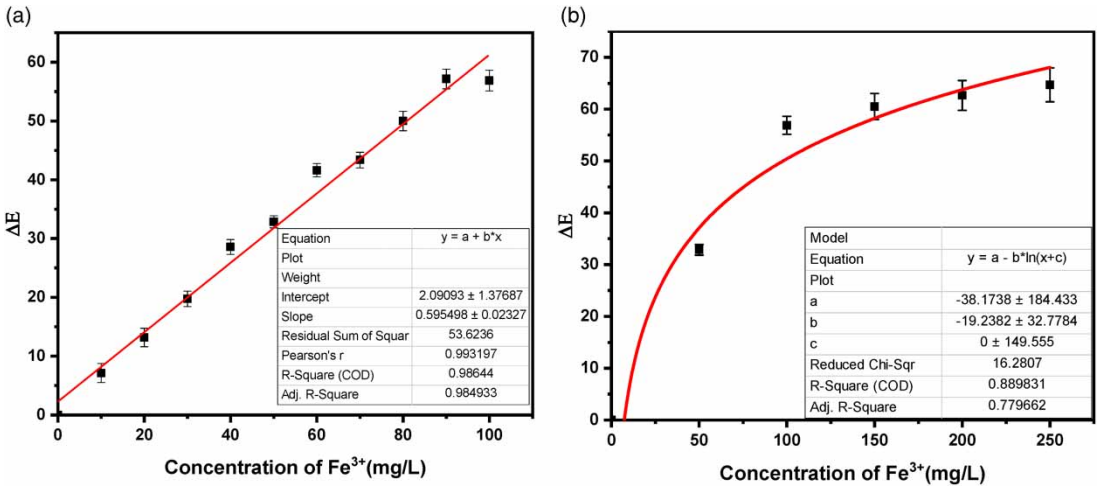


Figure 1 | The calibration curve for iron(III) quantitative image analysis. (a) 0–100 mg/L, and (b) 0–250 mg/L ($n = 3$).

Besides, iron also affects the color and pH value of water. Among the natural iron alum water samples, samples S1, S2, and S3 were preliminarily analyzed by flame absorption spectroscopy to determine the metal ion content (Table 3), where its iron content was 36.92, 34.02, and 26.45 mg/L, respectively. The results of the metal ion contents in natural iron alum water are relatively consistent with the results published in the previous work of our group (Nguyen *et al.* 2021). For the application of color indicator paper to determine iron(II) and iorn(III) contents in natural iron alum water samples, all ten water samples were carried out as indicated earlier. However, in natural iron alum water samples, iron(III) and iron (II) can exist simultaneously. Thus, on the surface of the iron indicator paper, there are two types of iron salts, $Fe(SCN)_3$ with blood red color (Reaction 1) and colorless $Fe(SCN)_2$ (Reaction 2). Therefore, when applying iron indicator paper combined with an image analysis method to determine iron content in natural iron alum water, only iron(III) content can be determined, while iron(II) content will not be analyzed. The disadvantage of this method in determining total iron (Fe^{2+} and Fe^{3+}) can be overcome by adding H_2O_2 solution at the rate of one drop of 30 wt.% H_2O_2 solution to 100 mL of iron alum water to oxidize all Fe^{2+} to Fe^{3+}. The determination results of the total iron content in 10 iron alum water samples by the color indicator paper method combined with the image analysis method are presented in Table 3. In particular, the determination results of iron content in natural iron alum water of 10 samples using the color indicator paper combined with the image analysis were very close to the iron content results analyzed by the flame absorption spectroscopy. From that, it can be said that this new analytical method has relatively good accuracy.

Besides image analysis methods, natural iron alum water samples were also performed with the colorimetric method with 1,10-phenanthroline reagent (according to TCVN 6177:1996 (ISO 6332:1988 E)), and the results are also presented in Table 3. The comparison results of iron analysis using these two methods with molecular absorption spectroscopy showed differences. Specifically, when comparing the results of 10 field samples, the new analysis method showed a difference of less than 5% compared to the molecular absorption spectroscopy method. The colorimetric analysis method using 1,10-phenanthroline reagent gives results that are always 10% greater than the molecular absorption spectroscopy method. This difference may be due to the existence of some components in natural iron alum water that reduce the ability to form complexes between iron ions and 1,10-phenanthroline reagents. The color change mechanism of the color indicator paper is shown in Figure 2. In addition, the recovery of the method (Equation (4)) tested with a given blank was 100%, and experimental testing of the test sample resulted in a recovery of about 109%. The concentration of the standard used to add to the test sample (field sample) was 32.090 mg/L (S), the field sample used was 43.041 mg/L (C), and the sample after the calibrating process was determined to be 78.110 mg/L (C_S).

With the principle of this analytical method, it can be said that this is a facile and convenient method to apply for the analysis of iron in natural iron alum water in the field. In addition, this method can be extended to determine Fe(III), Fe(II), and total iron easily with/without adding H_2O_2 in the pretreatment stage of natural iron alum water samples (Chen *et al.* 2021). Besides, when applying the image analysis method, the images need to be used

Table 3 | Results of iron analysis in natural iron alum water

	Results based on the color chart					TCVN 6177:1996 (ISO 6332:1988 E)			MEWW 3111B:2017
No.	1 Actual color of original water sample	2 Iron(III) concentration (mg Fe^{3+}/L)	3 Actual color after adding H_2O_2	4 Total iron concentration (mg/L)	5 Iron(II) concentration (mg Fe^{2+}/L)	6 mg Fe^{3+}/L	7 mg Fe^{2+}/L	8 Total iron concentration (mg/L)	Total iron concentration (mg/L)
S1		19.74 ± 0.79		36.49 ± 0.29	16.67	18.24	14.25 ± 0.42	32.49 ± 0.68	36.92 ± 0.85
S2		13.29 ± 0.81		34.76 ± 0.28	21.47	11.15	18.36 ± 0.28	29.51 ± 0.82	34.02 ± 1.14
S3		10.45 ± 0.34		25.92 ± 1.18	15.47	9.21	14.61 ± 0.18	23.82 ± 0.24	26.45 ± 1.32
S4		10.41 ± 0.55		23.8 ± 0.32	13.39	9.06	11.68 ± 0.22	20.74 ± 0.44	24.2 ± 1.08
S5		7.54 ± 0.19		18.04 ± 0.76	10.5	5.85	10.33 ± 0.45	16.18 ± 0.27	19.05 ± 1.20
S6		5.82 ± 1.0		16.47 ± 0.73	10.65	4.08	8.22 ± 0.32	12.3 ± 0.55	17.08 ± 1.04
S7		8.21 ± 0.77		14.04 ± 0.43	5.83	6.48	5.30 ± 0.28	11.87 ± 0.39	14.58 ± 0.74
S8		4.37 ± 0.29		12.86 ± 0.71	8.49	3.26	7.29 ± 0.44	10.55 ± 0.40	13.47 ± 0.65
S9		6.07 ± 0.53		9.5 ± 0.68	3.43	5.29	2.65 ± 0.26	7.94 ± 0.32	9.14 ± 0.76
S10		3.15 ± 53		9.53 ± 0.68	6.38	2.85	5.16 ± 0.27	8.01 ± 0.41	9.93 ± 0.52

Notes: Column (2) was calculated from the image analysis results of Column (1); Column (4) was calculated from the image analysis results of Column (3); Column (5) = Column (4) − Column (2); Column (6) = Column (8) − Column (7).

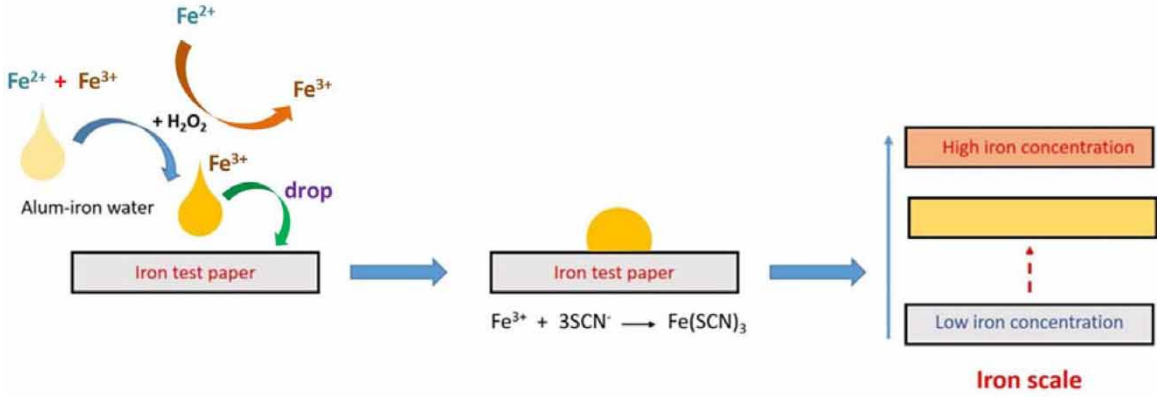


Figure 2 | Mechanism for the analysis of iron in natural iron alum water in the field.

on the same camera. The iron concentration in water when analyzing samples must not exceed 100 mg/L (exceeds the linear curve of the image analysis method), and iron must exist in ionic form in water. Water samples need to limit suspended sediment to avoid affecting the color of the test paper. In general, this method can be applied in the field and gives faster results than conventional analysis methods, which can save time and costs for the analysis process as well as avoid errors due to transportation, preservation, and handling of samples. In addition, this method can be applied to water samples with iron content below 100 mg/L. With an iron concentration greater than 100 mg/L, the dilution of the water sample before the analysis is considered.

4. CONCLUSIONS

The research has succeeded in producing iron indicator paper that is sufficiently sensitive to determine the presence and the content of ferric and ferrous ions in water. This method has created a color table according to changes in iron ion concentration, and the image analysis method has shown a linear standard curve: $\Delta E = 0.5955x + 2.0909$; $R^2 = 0.9864$, when iron concentration was less than 100 mg/L. When applied to natural iron alum water in the field, the image analysis method has shown analytical results close to the iron content results analyzed by flame absorption spectroscopy. At the same time, the method, with the combination of iron indicator paper and image quantification, proposed a fast and highly accurate method for quantifying iron content in natural iron alum water in the field. This is a useful and convenient method for many scientists, managers, engineers, and farmers in the assessment and classification of natural iron alum water in the locality. In addition, this method can be extended to quantify each type of Fe^{3+}, Fe^{2+}, and total iron ions for natural iron alum water.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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