Rapid determination of iron concentration in wastewater using color band formation

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Abstract

High concentrations of iron ions in water bodies cause odour and discolouration due to their precipitation. The total iron ion concentration is generally analysed on-site by forming coloured compounds and measuring colour intensity with spectrophotometric or visual analysis. In this work, we developed a detection tube as an alternative simple analytical method, where the total iron ion concentration is measured not by colour but by an objective parameter called the colour band length (CBL). Tiron was selected as the colouring agent since it oxidises divalent iron ions into a trivalent state and reacts with trivalent iron ions to form a 3:1 anion complex in alkaline media, giving a red colour. The coloured solution penetrates the detection tube by capillarity, and the colour band is formed by the entrapment of the hydrophobic ion pair of iron-tiron complex and quaternary ammonium ions of the adsorbent filled in the tube. The CBL was found to correlate well with the total iron ion concentration quantitatively under the optimized measurement condition, and 5 to 40 mg-FeL⁻¹ solutions could be measured with good objectivity and precision.

Keywords Anion complex · Capillarity · Colour band · Detection tube · Iron ion

1 Introduction

Iron is one of the most important metals used for structural materials, tools, and other equipment in the form of steel and/or alloys. Steel is an inexpensive and strong material, but it corrodes easily. Therefore, steel is coated with other metals and/or organic films to improve its corrosion resistance. In plating industries, wastewater containing iron is generated during the rinsing stage in the plating process, which involves acid. Iron is also one of the essential nutrients for humans; however, high concentrations of iron ions exert adverse effects on water such as smell and discolouration [8] and [13]. Iron exists as either divalent ion (Fe²⁺), trivalent ion (Fe³⁺) or iron complexes in water. Fe²⁺ ions

are easily oxidised into Fe³⁺ by the dissolved oxygen and precipitate as hydroxides, triggering the aforementioned problems. Hence, it is necessary to analyse the concentration of iron ions before discharging into natural water bodies. Water quality analysis is required for the operation of water treatment facilities. The official analytical methods for total iron ion concentration are spectrophotometric methods using o-phenanthroline, such as atomic absorption spectrometry (AAS), electro thermal atomic absorption spectrometry (ETAAS), and inductively coupled plasma atomic emission spectroscopy (ICP-AES) [3, 5, 7, 20]. The portable analytical tools are required for the on-site monitoring and maintenance of water facilities. Many analytical kits for total iron ion concentration analysis are

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developed and sold. These analytical tools employ absorption spectrophotometry or visual colourimetry. A relatively accurate measurement can be performed by absorption spectrometry although portable absorptiometers are relatively expensive. The simplest and most inexpensive method is visual analysis [18], which has the disadvantage of being inaccurate since visual judgment of colour tone lacks objectivity and can only determine the concentration discontinuously. It is also a problem that can easily be affected by individual differences and environmental factors like room brightness.

A method using colour band formation has been proposed to compensate for the drawbacks of visual analysis by individual differences due to naked eye observation. In this method, a coloured solution is allowed to penetrate a detection tube packed with adsorbents to form a coloured area (the colour band) resulting from the adsorption of coloured substances. Unlike conventional methods, the concentration is determined not from hue but from the colour band length (CBL). Since CBL is an objective parameter, the precision is expected to be improved by merely introducing a detection tube to the conventional colourimetric analysis. Compared to generally studied spectrophotometric methods with colorimetric sensors [17, 22] and/or portable instrument [12, 14], the detection tube is also inexpensive. Kiso and other researchers [1, 6, 9–11] have succeeded in developing this analysis method for phosphate ion, nitrite ion, ammonium ion, and arsenic ion detection; these detectable concentration ranges are 3–18 mgPO₄L⁻¹, 4–20 mg-NL⁻¹, 1–10 mg-NL⁻¹ and 0.01–0.1 mg-AsL⁻¹, respectively. Although the authors have succeeded in detecting these substances in domestic wastewater or drinking water with accuracy comparable to spectrophotometry using portable absorptiometry, the application has not been reported for the level of industrial wastewater analysis such as iron ion analysis yet.

In general, most published studies have reported not only on the colorimetric determination of low iron ions in the synthetic water condition without the interference of suspended solids but also the sorption of iron ion complexes using various kinds of sorbents with pH dependence [4, 15, 16, 19].

Focusing on developing countries with low cost and industrial plants with easy operation suffering from the damage of iron ions which require rapid detection easily, on the contrary, we established a colour band formation method using tiron as the colouring agent for measuring the total iron ions in aqueous solutions. Iron formed an anionic coloured complex with tiron, where charge was based on the number of tiron molecules bonded to the iron ion. Factors affecting the colour band formation, such as pH and amount of tiron added, were investigated. In addition, the correlation between the CBL and total iron ion

concentration and effect of co-existing metal ions under optimum measuring conditions were examined.

2 Materials and methods

2.1 Preparation of detection tube

The detection tube was prepared by packing an anion adsorbent into a transparent polypropylene column (3 mm (i.d.) × 150 mm (length)) which was lower end plugged with a melamine sponge. The adsorbent used was polyvinyl chloride (PVC) particles (particle size: 0.1 mm, Nacalai Tesque Inc., Kyoto, Japan) modified with benzylcetyldimethylammonium chloride (BCDMA, Tokyo Chemical Industry Co., Tokyo, Japan) through the following procedure: PVC and BCDMA were added together to a solution of methanol (Nacalai Tesque Inc., Kyoto, Japan) and shaken overnight. Thereafter, methanol was removed using a rotary evaporator and was subsequently dried at 50 °C overnight. The obtained adsorbent was stored in a desiccator prior to test. The BCDMA concentration in the adsorbent was 0.4% (w/w) and the bed height of the adsorbent was 130 mm.

2.2 Color development of iron ion solutions

Disodium 4,5-dihydroxybenzene-1,3-disulfonate monohydrate (C₆H₄Na₂O₈S₂·H₂O, Dojindo Laboratories, Kumamoto, Japan), commonly known as tiron, was selected as the colouring agent for iron ions. Tiron is a sulfonated catechol, as shown in Fig. 1. Since tiron is a kind of catechol, it has a high complex formation constant with Fe³⁺, and forms anionic complexes. The number of tiron molecules bonded to one Fe³⁺ changes with pH. One, two, and three tiron molecules can react with one Fe³⁺ at pH 1–4, 4–7, and above 7, respectively [21]. Additionally, in alkaline media, Fe²⁺ in the solution containing tiron is oxidised to Fe³⁺, so tiron can react with all the iron ions [2]. Tiron is relatively inexpensive and is thus suitable for simple analysis.

The procedure of colour development is shown in Fig. 2. Fe³⁺ and Fe²⁺ solutions were prepared using

Fig. 1 Structure of tiron

Fig. 2 Procedure of colour development

$\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ (Nacalai Tesque Inc., Kyoto, Japan) or $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ (Kishida Chemical Co, Osaka, Japan) as the iron source. A tiron solution was added to the iron ion containing solution and the solution pH was adjusted with a phosphate, acetate or carbonate buffer. The phosphate, acetate and carbonate buffers were prepared by mixing $(\text{NH}_4)_2\text{HPO}_4$ (Kishida Chemical Co, Osaka, Japan) with $\text{NH}_4\text{H}_2\text{PO}_4$ (Kishida Chemical Co, Osaka, Japan), CH_3COOH (Nacalai Tesque Inc., Kyoto, Japan) with CH_3COONa (Kishida Chemical Co, Osaka, Japan), and NaHCO_3 (Nacalai Tesque Inc., Kyoto, Japan) with Na_2CO_3 (Nacalai Tesque Inc., Kyoto, Japan), in appropriate ratios, respectively. The buffer was added after adding tiron to prevent the precipitation of iron hydroxide. Unless otherwise noted, the solution pH was adjusted from 8.5 to 9.5 with carbonate buffer. The solution was diluted to 20 mL with distilled water to obtain a final solution including iron ion and tiron of 5 to 40 mg-FeL^{-1} , and 0.18 to 18 mmolL^{-1} , respectively.

Co-existing metal ions were introduced by adding $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (Nacalai Tesque Inc., Kyoto, Japan), $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ (Nacalai Tesque Inc., Kyoto, Japan), $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ (Nacalai Tesque Inc., Kyoto, Japan), and $\text{MnSO}_4 \cdot 5\text{H}_2\text{O}$ (Nacalai Tesque Inc., Kyoto, Japan), respectively. Ethylenediaminetetraacetic acid (EDTA, Nacalai Tesque Inc., Kyoto, Japan) was tested as the masking agent. The co-existing metal ions and the masking agent were introduced before adding the tiron solution to the iron ion containing solutions.

Fig. 3 Absorption spectrum of the coloured solution

2.3 Measurement of CBL and the absorbance of the coloured solutions

The bottom end of the detection tube was immersed in the coloured solution. The coloured solution penetrated the tube by capillary action and reached the top of the tube in approximately 20 min. The immersion depth was about 5 mm. The coloured complex was adsorbed on the adsorbent during capillary action and formed a colour band. The CBL from the bottom was measured using a scale. When the boundary of the top of the colour band was inhomogeneous, the average length from the bottom to the top boundary was considered as the CBL. The absorbance of the coloured solution was measured using a UV-vis spectrophotometer (UV 2450, Shimadzu, Japan).

3 Results and discussion

3.1 Formation of coloured complex

Fe^{3+} forms a complex with tiron and the coordination number changes according to the pH of the solution. Fe^{3+} forms one coordinate bond with one tiron below pH 4, with two tiron between pH 4 and 7, and with three tiron over pH 7. The negative charge of the complex reaches a maximum of -9 at three-fold coordination. Moreover, the colour changes along with the change in pH: blue below pH 4, purple from pH 5 to 7, and red over pH 7. Figure 3 shows the absorption spectrum of the coloured solution at pH 4.0, 5.5, and 8.5 adjusted using acetate, phosphate and carbonate buffer, respectively. The maximum absorption wavelengths at pH 4.0, 5.5, and 8.5 are 600, 510, and 480 nm, respectively. The maximum absorption wavelength decreased, while the absorbance at each maximum

Fig. 4 Absorption spectrum of the coloured solution

absorption wavelength increased with an increase in solution pH. It was suggested that the alkaline region is suitable to apply this method because the complex that forms in this region has a large negative charge and absorbance.

Fe^{2+} does not form any complex with tiron; however, the same complex was formed in alkaline media containing Fe^{2+} (Fig. 4). It is reported that the much greater stability of the Fe^{3+} complex compared to that of Fe^{2+} resulted in the lowering of the reduction potential, and oxygen immediately oxidised the Fe^{2+} ion into Fe^{3+} [2]. In addition, in the optimum conditions described later, almost the same colour bands were obtained regardless of the valence of the iron ions. Therefore, this method can be used to analyse the total iron ion concentration.

3.2 Effect of tiron concentration

The amount of tiron is expected to affect the formation of the iron complex and the CBL. Hence, the amount of tiron was varied from 1 to 100 times (0.18 to 18 mmolL^{-1}) against that of the iron ions of 10 mg/L-Fe ($0.18 \text{ mmol-FeL}^{-1}$) and its effect on the iron complex formation and CBL was investigated. The absorbance at 480 nm , which corresponds to the 3:1 complex of tiron and Fe^{3+} , is shown in Fig. 5. The result evidenced the formation of the 3:1 complex of tiron and Fe^{3+} and the necessity to add thrice more tiron than Fe^{3+} to complete the complex formation. When excess tiron was added to the system, unreacted tiron remained. The effect of the amount of unreacted tiron on CBL is shown in Fig. 6. The CBL showed a constant value when the amount of the unreacted tiron was between 1.6 to 6.0 mmolL^{-1} ; however, the CBL increased when the amount of the unreacted tiron was below 1.6 mmolL^{-1} or more than 6.0 mmolL^{-1} . The colour band became lighter in both cases. The reason for the stretching in CBL can be

Fig. 5 Effect of tiron ratio on absorbance

attributed to the following: BCDMA and tiron are known to form a hydrophobic ion pair. When the amount of unreacted tiron is below 1.6 mmolL^{-1} , the BCDMA must have partially dissolved in the solution due to the insufficient formation of hydrophobic ion pairs. On the contrary, when the amount of unreacted tiron is more than 6.0 mmolL^{-1} , the reaction between the excess unreacted tiron and BCDMA becomes competitive, and that between the iron-tiron complex and BCDMA is avoided. Therefore, the iron-tiron complex must have migrated to a higher position in the tube, resulting in an increased CBL. The optimum range is 1.6 to 6.0 mmolL^{-1} unreacted tiron, because the dissolution of BCDMA was suppressed, and the chelate preferentially reacted with BCDMA. To detect an iron ion concentration of around 10 mgL^{-1} , the concentration of the tiron solution was decided to be 5.4 mmolL^{-1} . In the measurement range of this method described later (5 to 40 mg-FeL^{-1}), the unreacted tiron concentration falls within the optimum range.

3.3 Effect of pH

The pH of the coloured solution was varied between 7 and 9 using a phosphate or carbonate buffer, and the influence on colour development and colour band formation was investigated. The constant absorption spectrum suggested that tiron reacts with almost all Fe^{3+} to form a 3:1 anion complex above pH 7. In addition, the iron-tiron complex adsorbed onto the adsorbent well for pH 8.5–9.5, and the CBL showed almost a constant value. In this study, the subsequent experiments were conducted from pH 8.5 to 9.5. Because the buffer type had no influence, carbonate buffer, which was relatively harmless, was used in subsequent experiments.

Fig. 6 Effect of unreacted tiron concentration on CBL. **a** CBL increased as the unreacted tiron was below 1.6mmolL^{-1} or more than 6.0mmolL^{-1} , **b** corresponding CBL testing photos

Fig. 7 Time profiles of absorbance and CBL

3.4 Stability of the coloured solution

The time profiles of the absorbance at 480 nm and the corresponding CBLs were investigated to assess the stability of the complex. The profiles are shown in Fig. 7. A part of the unreacted tiron in the solution was oxidised to yellow quinone by the dissolved oxygen and the absorbance increased gradually. The coloured solution was introduced into the detection tube, which already contained some oxidised tiron after 180 min of preparation. A different thin yellow band was formed on the upper part of the colour band of the complex. The adsorption of oxidised tiron to the adsorbent tube was weaker than that of the iron-tiron complex. It is considered that oxidised tiron has a negligible effect on CBL measurement and the stable measured value can be obtained at least 180 min after preparation by this method.

Table 1 Effects of competitive cation for CBL

Competitive cation	Error (%)
Cu^{2+}	8.5
Ni^{2+}	1.7
Zn^{2+}	-1.7
Mn^{2+}	487
Mn^{2+} with EDTA	1.7

Error [%]: $100 \{ (\text{CBL with competitive cation}) - (\text{CBL without competitive cation}) / (\text{CBL without competitive cation}) \}$

3.5 Effects of co-existing metal ions

Co-existing metal cations (100mgL^{-1}) were added to a coloured solution containing 10mg-FeL^{-1} , and their influence on CBL was investigated as shown in Table 1. Copper, nickel, and zinc, which are the main cations used in the plating of steel, did not influence the CBL; however, manganese exhibited large positive errors. It is considered that tiron reacted with not only Fe^{3+} but also manganese ions. The effect of manganese ions could be restrained by the addition of EDTA as a masking agent before the addition of tiron. EDTA also formed a complex with the iron ions; however, colour development was not influenced due to a higher stability constant between tiron and Fe^{3+} under this condition.

3.6 Measurement of iron ion concentration using the developed detection tube

The relation between the iron ion concentration and CBL along with the pictures of colour bands are shown in Figs. 8 and 9, respectively.

Fig. 8 Calibration plot of iron ion. **a** overall view, **b** enlarged view. Error bar: standard deviation

Fig. 9 Colour bands formed in detection tubes

From previous experiments, the iron concentration and pH of the coloured solutions were adjusted to 5.4 mmol L^{-1} , and 8.5 to 9.5, respectively. Measurements were taken 3 times in each concentration, and the measurement time was about 20 min regardless of the concentration. In the range of 5 to 40 mg/L-Fe, the iron ion concentration and CBL had a good linear relationship. It is considered that this method is applicable within this concentration range. At a low iron ion concentration, the boundary of the coloured band becomes blurred, and the measurement accuracy is considered to be lower. The relative standard deviation was 5.6% at maximum (14 mg-Fe L^{-1}). The result indicated that the iron ion concentration could be analysed with high accuracy compared to the pack test, which is one of standard analytical method in Japan.

As shown in Fig. 10, the CBL has more precisely approached to the standard value than pack test during the 13 times of control experiments.

The coloured solution was also prepared by adding a certain amount of colouring agent and pH buffering agent in powder form to the iron ion solution sample. The influence on the absorbance and CBL due to the change in volume upon reagent dissolution was investigated. As a result, there was almost no change in absorbance,

Fig. 10 The comparison of experimental results of 13 times between Color band length method developed in this work and Colorimetric method

therefore, the volume increase of the sample due to dissolution of the powder was negligible. From Fig. 8, CBL also shows the same value as the conventional preparation method.

4 Conclusions

Compared to commonly studied visual determination of iron ions, we have developed a simple and highly accurate analytical method for the determination of the total iron ion concentration by colour band formation. The optimal measurement conditions were to use a tiron solution of 5.4 mmol L⁻¹ and pH of 8.5 to 9.5. A coloured solution containing 5 to 40 mg-Fe L⁻¹ could be determined by this method. The coloured solution was stable for at least 180 min. The total concentration of Fe²⁺ and Fe³⁺ could be measured without adding an oxidiser or a reducing agent. Moreover, it was found that the influence of competitive cations expected in wastewater, generated after plating on steel, could be avoided. The measurement time was about 20 min and the relative standard deviation was less than 5.6%. This method is expected to be promising method for practical applications especially in high concentration of iron wastewaters.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interests.

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