

Determination of Iron(III) by Room Temperature Ionic Liquids/Surfactant Sensitized Fluorescence Quenching Method

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Abstract A new method for the determination of iron (III) has been described. The analytical procedure was based on the fluorescence quenching of salicylfluorone (SAF) by iron (III) and the fluorescence quenching value (ΔF) could be increased in the medium of room temperature ionic liquids (RTILs) (1-ethyl-3-methylimidazolium ethyl sulfate)/SDS (sodium dodecyl sulfate). The main factors influencing the fluorescence quenching (ΔF) were investigated in detail. Under the optimal conditions, the linear equation was $\Delta F = 319c + 176.5$ ($c: \mu\text{g}\cdot\text{mL}^{-1}$, $r=0.9936$). The linear range of calibration curve was $0.2\text{--}1.1 \mu\text{g}\cdot\text{mL}^{-1}$ and the detection limit was $8.3 \text{ ng}\cdot\text{mL}^{-1}$. The preliminary sensitized mechanism was discussed with distribution coefficient and fluorescence quantum yield in different media. The method has been applied to the determination of Fe (III) in water samples with satisfactory results.

Keywords Iron (III) · Room temperature ionic liquids (RTILs) · SDS micelle · Salicylfluorone (SAF) · Fluorescence quenching method

Introduction

Iron has an important physiological functions in the human body and responsible for oxygen delivery between the blood and tissue. People who lack of iron will be suffering from iron

deficiency anemia and cause a serious impact on human health. Water pollution of iron mainly is ore dressing, smelting, mechanical processing, industrial plating and pickling waste water [1]. Iron has been widely used in human daily life and widespread in the environment. Hence, the determination of iron in the sample analysis is very significant.

Until now, the methods determination iron were chromatography [2], electrochemical methods [3] and spectroscopic method [4–8] (including UV spectroscopy [4], spectrofluorimetry [5, 6], atomic absorption spectrometry [7] and flow injection spectrometry [8]). Spectrofluorimetry for determination of Fe (III) have been based on quenching phenomena [5, 6].

The sensitivity of spectral analysis could be enhanced in suitable medium (such as surfactant, β -CD, room temperature ionic liquids) [9–15]. In our previous publications, the sensitizing effect of surfactant on the determination of metal ion by ultraviolet spectrometry and spectrofluorimetry were developed [9–13]. We have reported the determination of trace nickel in nonionic microemulsion medium by the fluorescence quenching method with satisfactory results [14]. Room temperature ionic liquids (RTILs) which were proposed as greener alternatives to volatile organic solvents received much attention in analytical chemistry such as electrochemistry analysis and chromatography and spectrum analysis [15–17]. In spectral analysis, RTILs mainly used as sensitizer [15] and extractant [16, 17].

RTILs and surfactants could form an ordered molecular assembly which not only own the inherent of surfactant but also possess many advantages of RTILs, and achieve the integration of their nature [18]. The RTILs/surfactant (ionic liquid 1-butyl-3-methylimidazolium chloride/Triton X-100 micelle) as a novel sensitizer for spectrophotometric determination aluminum had been reported by Guopo Zhao [15]. But the sensitized effect of RTILs/surfactant on spectro-

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fluorimetry seems to be lacking. In this paper, RTILs/SDS has been applied as sensitizer for the determination of Fe (III). A preliminary study of the sensitizing mechanism was discussed with distribution coefficient and fluorescence quantum yield. The proposed approach was evaluated by analysis of water samples with satisfactory results.

Experimental

Instrumentation and Reagents

A Hitachi F-4500 spectrofluorimeter (Japan) was used for all the fluorescence measurement, with excitation and emission slits at 5.0 nm, $\lambda_{ex}=365$ nm. The pH of solutions was measured using a pHS-25 pH meter (Shanghai, China). DDS-11A digital conductivity instrument (Shanghai, China). All absorption spectral recordings and absorbance measurements were performed on a UV-723PC spectrophotometer (Shanghai, China). 2K-82 B vacuum drying oven (Shanghai, China).

A standard Fe (III) stock solution of $1000.0 \mu\text{g}\cdot\text{mL}^{-1}$ was prepared. The solution was diluted to $10.0 \mu\text{g}\cdot\text{mL}^{-1}$. Series of HAc-NaAc buffer solutions, pH 2.0 to 5.0. $1 \times 10^{-4} \text{ mol}\cdot\text{L}^{-1}$ solution of SAF, 10% solution of SDS and 10% solution of ionic liquid (1-Ethyl-3-methylimidazolium ethyl sulfate) was prepared. A standard quinine sulfate stock solution of $1.00 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$ was prepared. The solution was diluted as needed to concentration by $0.050 \text{ mol}\cdot\text{L}^{-1} \text{ H}_2\text{SO}_4$. All reagents used were of analytical grade and their solutions were prepared by serial dilutions with distilled water.

Procedure

Synthesis Method of RTILs (1-Ethyl-3-Methylimidazolium Ethyl Sulfate)

Synthesis according to literature [19, 20].

Fluorescence Spectrum

0.50 mL $10.0 \mu\text{g}\cdot\text{mL}^{-1}$ Fe (III) solution, 1.00 mL pH=3.00 buffer solution, 1.00 mL $1 \times 10^{-4} \text{ mol}\cdot\text{L}^{-1}$ SAF, 1.20 mL 10% SDS and 2.00 mL RTILs 10% were transferred into 10.0 mL volumetric flask. The mixtures were diluted to the mark with water and shaken thoroughly. Then the fluorescence intensity was measured at $\lambda_{ex}/\lambda_{em}=365/520$ nm against a reagent blank.

Sample Preparation

For sample analysis, samples (GSBZ50019-90) were diluted 250-fold with water and 2.0 mL sample solution

were chosen to determine the fluorescence intensity experiment.

Determination of Critical Micelle Concentration (cmc) [21]

In this work, cmc values of the medium were measured by conductivity measurements. The conductivity cell was calibrated with solutions in the appropriate concentration range. The measurement was started with a dilute solution and the subsequent concentrated solutions were prepared by adding a previously prepared stock solution. Various concentrations of solutions were prepared in the range 0–20 $\text{mmol}\cdot\text{L}^{-1}$. The conductivity of these solutions was measured at room temperature.

Determine of Distribution Coefficient [22]

The distribution coefficients (K_D) of SAF in different medium were measured. 1.00 mL pH=3.00 buffer solution, 1.00 mL $1.0 \times 10^{-4} \text{ mol}\cdot\text{L}^{-1}$ SAF, various amounts of SDS and RTILs were added to 10.0 mL volumetric flask. Selected some appropriate systems and drew the absorption curve under 25 °C at different concentration of surfactant.

K_D was calculated as follows:

$$\frac{1}{E_{\psi} - E_W} = \frac{1}{K_D(E_m - E_W)} \times \frac{1}{c_D} + \frac{1}{E_m - E_W}$$

Briefly, E_{ψ} was the apparent molar absorptivity of ligand under certain concentration of SDS (or RTILs), E_w and E_m were the apparent molar absorptivity of ligand in different medium, and C_D was the concentration of SDS (or RTILs).

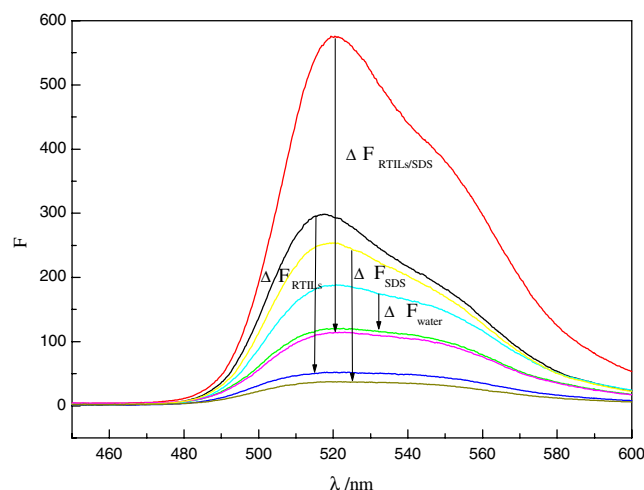


Fig. 1 Fluorescence spectrum in different media. ($c_{\text{Fe(III)}} = 0.50 \mu\text{g}\cdot\text{mL}^{-1}$)

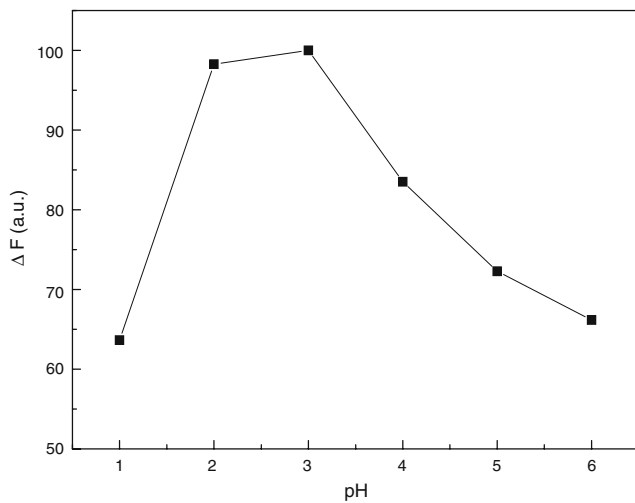


Fig. 2 Effect of pH on ΔF. Conditions :c(Fe³⁺), 0.50 μg·mL⁻¹; SAF, 1×10⁻⁵ mol·L⁻¹; SDS, 1.2%; RTILs, 2.0%

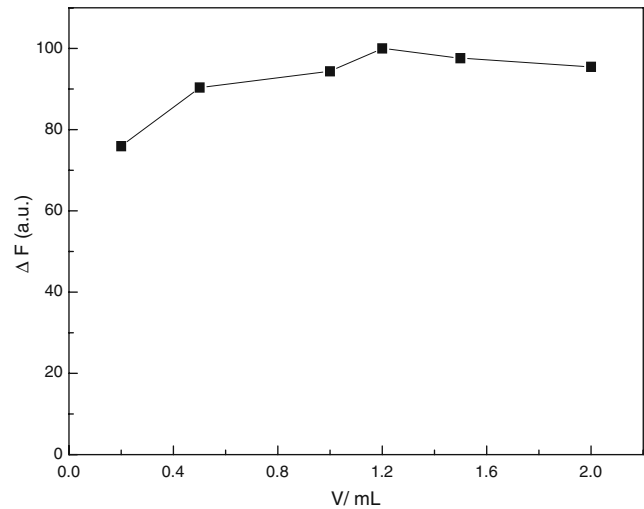


Fig. 4 Effect of amount of SDS on ΔF. Conditions :c c_{Fe(III)}, 0.50 μg·mL⁻¹; SAF, 1×10⁻⁵ mol·L⁻¹; RTILs, 2.0%

K_D in different medium could be found by plotting 1/E_ψ - E_w with 1/c_D.

and n_s are the refractive index of the standard and unknown, and Φ_S=0.546 (25 °C) is known.

Determination of Relative Fluorescence Quantum Yield [23]

Fluorescence quantum yields of SAF were measured by ration value of integrated area under corrected fluorescence spectra in the various media. Quinine sulfate was applied to the standard. According to the equation

$$\Phi_f = \frac{n_f^2}{n_s^2} \cdot \frac{D_f}{D_s} \cdot \Phi_s$$

Φ_S and Φ_f are corresponding the standard and unknown fluorescence quantum yield, and D_S and D_f are the integral areas of two calibration fluorescence emission curves, n_f

Results and Discussion

Fluorescence Spectra

In the experiment, various medium were chosen for discussing their effects on the fluorescence intensity quenching value (ΔF), and all results were shown in Fig. 1. From Fig. 1, the order of ΔF in different medium was as follows: ΔF_{RTILs/SDS}>ΔF_{RTILs}>ΔF_{SDS}>ΔF_{water}. So, RTILs/SDS possessed the highest sensitivity in corresponding other mediums and was chosen as the medium.

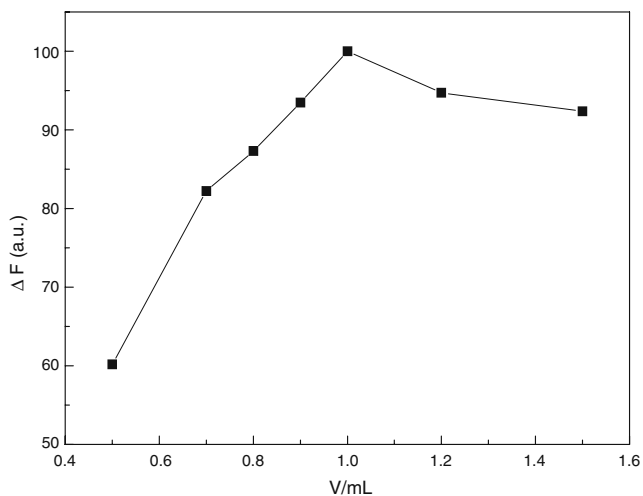


Fig. 3 Effect of amount of SAF on ΔF. Conditions :c c_{Fe(III)}, 0.50 μg·mL⁻¹; SDS, 1.2%; RTILs, 2.0%

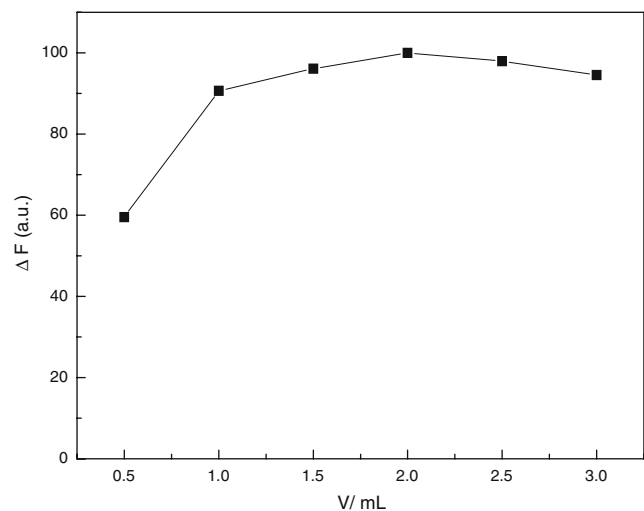


Fig. 5 Effect of amount of RTILs on ΔF. Conditions :c c_{Fe(III)}, 0.50 μg·mL⁻¹; SAF, 1×10⁻⁵ mol·L⁻¹; SDS, 1.2%

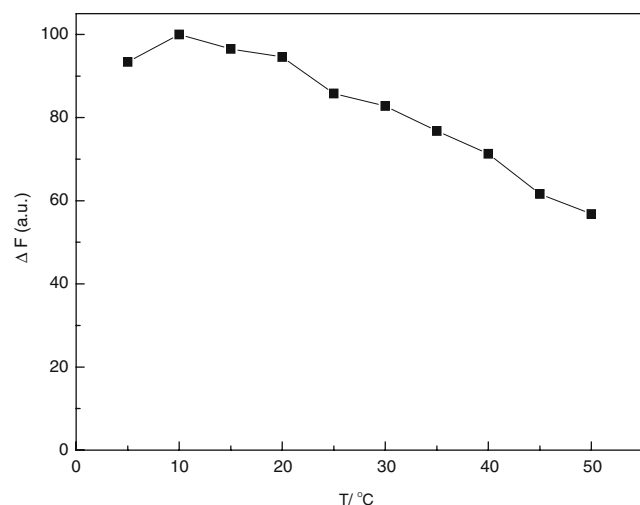


Fig. 6 Effect of temperature Conditions :c $c_{\text{Fe(III)}}$, $0.50 \mu\text{g}\cdot\text{mL}^{-1}$; SAF, $1 \times 10^{-5} \text{ mol}\cdot\text{L}^{-1}$; SDS, 1.2%; RTILs, 2.0%

Effect of pH

The effect of pH on ΔF was studied. The result was summarized in Fig. 2. When the value of pH varied in range of 1.00–6.00, the quenching value ΔF reached the maximum at pH=3.00. However, when pH was increased, ΔF declined rapidly. The possible reason was that the formation and the stability of Fe(III)-SAF could be influenced by the system acidity. Thus, 1.00 mL buffer solution of pH=3.00 was selected for further studies.

Effect of Amount of SAF

Amounts of SAF solution were added variously to investigate the effects on ΔF (Fig. 3). The fluorescence intensity

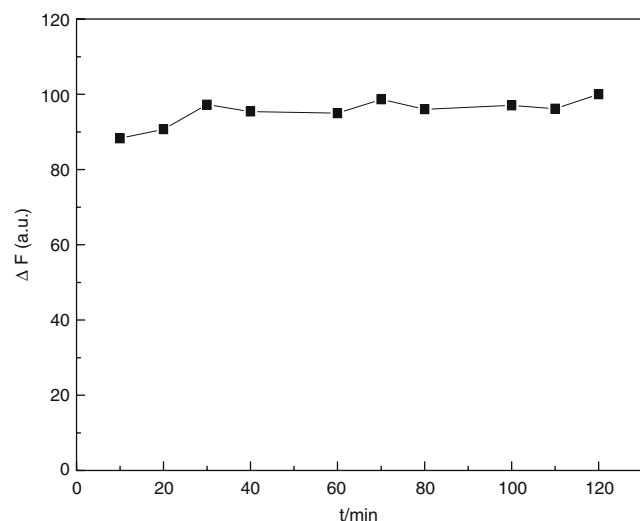


Fig. 7 Effect of time Conditions :c $c_{\text{Fe(III)}}$, $0.50 \mu\text{g}\cdot\text{mL}^{-1}$; SAF, $1 \times 10^{-5} \text{ mol}\cdot\text{L}^{-1}$; SDS, 1.2%; RTILs, 2.0%

Table 1 Effect of foreign ions on determination of iron (III) ($c_{\text{Fe(III)}}$: $0.50 \mu\text{g}\cdot\text{mL}^{-1}$)

Interferences ions	Tolerance ratio (w_1/w)
K^+ , Mg^{2+} , SO_4^{2-} , NO_3^- , Cl^- , Na^+	1000
Cu^{2+} , Mn^{2+}	500
Ca^{2+}	200
Zn^{2+}	50
Al^{3+} , Ac^-	5

quenching value ΔF enhanced with the amount of SAF and reached a maximum value for SAF volume of 1.00 mL. When the amount of SAF was higher than 1.00 mL, ΔF decreased. This may be caused by self-quenching of SAF at higher concentration ($F_{\text{SAF}} \downarrow$, $\Delta F = F_{\text{SAF}} - F_{\text{Fe-SAF}} \downarrow$). Hence, 1.00 mL $1.00 \times 10^{-4} \text{ mol}\cdot\text{L}^{-1}$ SAF was chosen as optimum for further studies.

Effect of Amount of SDS

In the experiment, various amounts of SDS were added to investigate the effects on (Fig. 4). With the increase of volume of SDS, the fluorescence intensity quenching value ΔF increased gradually and achieved maximum when the volume approached to 1.20 mL. As a result, 1.20 mL 10% SDS was selected in this work.

Effect of Amount of RTILs

The influence of RTILs on the quenching value ΔF was investigated by changing the amount of RTILs (Fig. 5). It was found that ΔF was appropriate for maximum and keep unchanged when the volume of RTILs was 1.00–3.00 mL. So, optimum volume of RTILs chosen for this work was 2.00 mL.

Effect of Temperature

Temperature is an important factor in spectrofluorimetry. The effect of temperature was tested in 5–55 °C (Fig. 6). In the medium of RTILs/SDS, ΔF unchanged when the temperature was increased in the range of 5–20 °C and gradually decreased when $T > 20$ °C. Because the fluorescence intensity of SAF lowered slowly with the increase of temperature ($F_{\text{SAF}} \downarrow$, $\Delta F = F_{\text{SAF}} - F_{\text{Fe-SAF}} \downarrow$). So, 20 °C was chosen for this work.

Effect of Time

The effect of time on the quenching value ΔF was studied (Fig. 7). At room temperature, the quenching reaction could reach equilibrium immediately, and the ΔF varied very little in almost 2 h which could show a good stability of the system.

Table 2 Determination of standard substance ($n=5$)

GSBZ50019-90 ^a	Criterion (µg/mL)	Determination (µg/mL)	RSD (%)
	1.92±0.13	1.98±0.07	4.26

^a After suitable dilution

Effect of Interfering Ions

The interference of different foreign substrates were discussed in the determination of $0.50 \mu\text{g}\cdot\text{mL}^{-1} \text{Fe}^{3+}$. With a relative error of less than $\pm 5\%$, the tolerance limits for various foreign ions were listed in Table 1. It was showed that most of metal ion did not influence the determination of Fe^{3+} . So the method possessed a good selectivity.

Analytical Parameters

The calibration graph for the determination of iron (III) was obtained under the best experimental conditions. The results showed a good linear relationship was observed over the range of $0.20\text{--}1.10 \mu\text{g}\cdot\text{mL}^{-1}$. The linear equation was $\Delta F=319c+176.5 (\mu\text{g}\cdot\text{mL}^{-1})$, $r=0.9936$. The detection limit of iron (III) was determined to be $8.3 \text{ ng}\cdot\text{mL}^{-1}$. The relative standard deviations (R.S.D.) was $4.26\% (n=5)$.

Samples Determination

The proposed method was applied for the determination of Fe^{3+} in standard reference materials and in lake water samples. The standard addition method was used and the analytical results and the recovery are presented in Tables 2 and 3. The average recoveries of the proposed method were $96.0\%\text{--}103.0\%$.

Discussion of Mechanism of Sensitizing Effect

The sensitizing effect of the RTILs/surfactants on spectroanalysis rested with two factors: (1) the solubilization capacity of medium; (2) the microenvironment of medium [11]. In this paper, the discussion of mechanism of sensitizing effect was follow as (1) the formation of the ordered molecular assembly, which interfere on the solubilization capacity of medium; (2) the effect of microenvironment of medium on the fluorescence quantum yield of SAF

The Formation of the Ordered Molecular Assembly

There were mutual influences which could reflect on the change of critical micelle concentration (cmc) between the RTILs and surfactants. The cmc decreased when hydrophobic RTILs soluble in surfactant and increased when hydrophilic RTILs interact with the surfactant in the aqueous phase [24].

In this work, conductivity measurements were used to determine the critical micelle concentration (cmc). The cmc was obtained from the intersection point of the straight lines of pre- and post-micellar concentration range [21]. The plots of electrical conductivity in the different concentration of the solution were shown in Fig. 8. The concentration corresponding to the inflection point was taken as the cmc. The results were listed in Table 4.

As can be seen from Table 1 that the cmc of SDS, RTILs and SDS/RTILs were $8.4 \text{ mmol}\cdot\text{L}^{-1}$, $3.0\text{--}4.0 \text{ mmol}\cdot\text{L}^{-1}$ and $11.0\text{--}12.0 \text{ mmol}\cdot\text{L}^{-1}$, respectively. There was discrepancy for the cmc of the mixed solution (SDS/RTILs) in determination value and the Clint model value[25]. On the assumption of ideal mixing behavior, the model could be used to calculate the cmc of the mixed medium. The critical micelle concentration of the mixed system are given by

$$\frac{1}{\text{cmc}^*} = \frac{\alpha_1}{\text{cmc}_1} + \frac{1 - \alpha_1}{\text{cmc}_2}$$

(where α_1 was the mole fraction of 1 in mixed medium; cmc_1 , cmc_2 , cmc^* were the cmc of single and mixed medium, respectively). In this work, the molar ratio of SDS and ionic liquid was 1:2. So, the cmc of the mixed solution should be within $3.8\text{--}4.8 \text{ mmol}\cdot\text{L}^{-1}$. The result demonstrated that the mixed medium could form a new ordered molecular assembly. The sensitivity of the determination could be improved by the ordered molecular assembly.

Table 3 Analysis of water samples

Sample	Added (µg/mL)	Determined (µg/mL)	Recovery (%)
Lake water ^b	0.00	0.175	
	0.20	0.360	96.0
	0.30	0.489	103.0
	0.40	0.556	96.7
	0.50	0.653	96.7

^b The lake water was condensed by 3 times

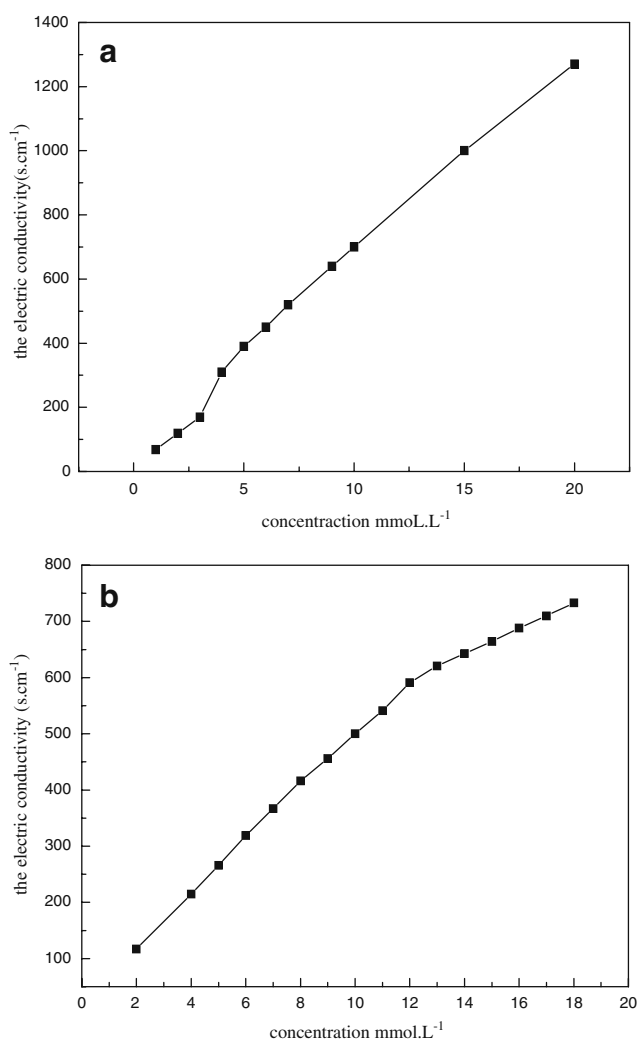


Fig. 8 The plots of electrical conductivity in the different concentration. **a** Electrical conductivity vs. the concentration of RTILs. **b** Electrical conductivity vs. the concentration of SDS/RTILs

Distribution Coefficient (K_D) and Fluorescence Quantum Yield (Φ_f) in Different Medium

The fluorescent agents, the dissolution in the medium and microenvironment of the external medium can directly influence the F_{SAF} . Distribution coefficient (K_D) could be

Table 4 cmc of the different medium

Medium	cmc (mmol/L)	
	Determination value	Clint model value
SDS	8.4 ^c	
RTILs	3.0–4.0	
SDS/RTILs(1:2)	11.0–12.0	3.8–4.8 ^d

^c The literature [24]; ^d calculate by Clint model[24]

Table 5 Distribution coefficient (K_D) in different medium

Medium	K_D
SDS	127.61
RTILs	99.15
SDS/RTILs	178.37

used to research the solubilization of fluorescent agent in the medium. K_D values in different media were listed in Table 5.

As can be seen from Table 5, K_D could reach the maximum in mixed media. In single medium (SDS or RTILs), fluorescent agent is mainly dispersed in small droplets, the solubility and distribution coefficient will increase. In mixed medium (RTILs/SDS), the palisade tissue of the system is the role structure. Under the effect of the electrostatic force, the presence of cations in ionic liquids could change the distance of the palisade tissue. Part of the distance increases and the others decrease. Compared with the single medium (SDS or RTILs), more micelle molecular are accommodated in palisade tissue where the distance increase. It can enhance the solubility of fluorescent agent in medium. So, the order molecular assembly could increase the sensitivity of fluorescence spectrophotometry.

The fluorescence quantum yields was one of the mostly basic and significant parameters in all the characters of fluorescence substance [11]. It was represented the ability of translating absorption energy to fluorescence. The Φ_f value was tightly related to the internal environment (the fluorescent agents) and the external environment (the microenvironment of medium). In our research, SAF was chosen as the fluorescent agents. So, the fluorescence quantum yields of SAF changed with the inflect of the microenvironment of medium. The fluorescence quantum yields of SAF (Φ_f) in various media were determined respectively. The results were listed in Table 6.

As show in Table 6, the order of fluorescence quantum yield (Φ_f) in different medium was $\Phi_{RTILs/SDS} > \Phi_{SDS} > \Phi_{RTILs} > \Phi_{water}$. It is the same with their sensitivities. The changes could be considered in internal and external environment. Compared with the single medium (SDS or RTILs), the external environment provided by the mixed media (RTILs/SDS) is more favorable on solubilization. Moreover, the microenvironment which was formed by

Table 6 Fluorescence quantum yield (Φ_f)

Medium	SAF Φ_f
water	0.01057
SDS	0.02456
RTILs	0.08017
RTILs/SDS	0.1127

RTILs and micelle could offer the protective environment to the excited single state and could reduce the non-radiative of the fluorescent agent. The mixed medium can form the most suitable environment to improve system sensitivity. As a result, fluorescence quantum yield in mixed media is the largest.

Conclusion

The sensitizing effect of the RTILs/SDS on determination of Fe (III)-SAF was developed in this paper. The main factors affecting the determination were investigated in detail. The formation of the ordered molecular assembly by measuring the cmc of the medium, the distribution coefficient and fluorescence quantum yield of SAF in various mediums were used to discuss the mechanism of sensitizing effect. The proposed method has been applied satisfactorily to determine Fe (III) in environmental reference materials and lake water.

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