Available online at www.sciencedirect.com
SCIENCE DIRECT®
Talanta 65 (2005) 525–530
Talanta
www.elsevier.com/locate/talanta

Cloud point extraction of iron(III) and vanadium(V) using 8-quinolinol derivatives and Triton X-100 and determination of 10^{-7} mol dm^{-3} level iron(III) in riverine water reference by a graphite furnace atomic absorption spectroscopy

Akira Ohashi*, Hiromi Ito, Chikako Kanai, Hisanori Imura, Kousaburo Ohashi

Department of Environmental Sciences, Faculty of Science, Ibaraki University, Mito 310-8512, Japan

Received 5 April 2004; received in revised form 8 July 2004; accepted 14 July 2004
Available online 26 August 2004

Abstract

The cloud point extraction behavior of iron(III) and vanadium(V) using 8-quinolinol derivatives (HA) such as 8-quinolinol (HQ), 2-methyl-8-quinolinol (HMQ), 5-butyloxymethyl-8-quinolinol (HO_4Q), 5-hexyloxymethyl-8-quinolinol (HO_6Q), and 2-methyl-5-octyloxymethyl-8-quinolinol (HMO_8Q) and Triton X-100 solution was investigated. Iron(III) was extracted with HA and 4% (v/v) Triton X-100 in the pH range of 1.70–5.44. Above pH 4.0, more than 95% of iron(III) was extracted with HQ, HMQ, and HMO_8Q. Vanadium(V) was also extracted with HA and 4% (v/v) Triton X-100 in the pH range of 2.07–5.00, and the extractability increased in the following order of HMQ < HQ < HO_4Q < HO_6Q. The cloud point extraction was applied to the determination of iron(III) in the riverine water reference by a graphite furnace atomic absorption spectroscopy. When 1.25×10^{-3} M HMQ and 1% (v/v) Triton X-100 were used, the found values showed a good agreement with the certified ones within the 2% of the R.S.D. Moreover, the effect of an alkyl group on the solubility of 5-alkyloxymethyl-8-quinolinol and 2-methyl-5-alkyloxymethyl-8-quinolinol in 4% (v/v) Triton X-100 at 25 °C was also investigated.

Keywords: Cloud point extraction; Iron(III); Vanadium(V); 8-Quinolinol derivative; Triton X-100; Graphite furnace atomic absorption spectroscopy

1. Introduction

Determination of trace metal ions in natural water samples is interesting and important for analytical chemistry. To detect them, we can use several methods such as atomic absorption spectroscopy, inductively coupled plasma atomic emission spectrometry, and so on. However, it is difficult to determine trace metals in their samples directly, because of their low concentration and coexistence of matrix species. Therefore, pre-concentration and separation of analyte are needed before measuring.

The most common procedures for the pre-concentration of analytes interested are the use of either liquid–liquid or solid sorbents extraction. Recently, a cloud point extraction of organic and inorganic compounds using non-ionic surfactants have been concerned in analytical chemistry [1–6]. On heating a surfactant solution over a critical temperature, the solution easily separates into two distinct phases. The one contains a surfactant at a concentration below, or equal to, a critical micelle concentration. The other is a surfactant-rich phase. An analyte can be pre-concentrated with a large degree of the concentration into a surfactant-rich phase. The cloud point extraction has some advantages such as inexpensive, good concentration efficiency, environmentally lower toxicity, and small environmental pollution over conventional liquid–liquid extraction.

Watanabe et al. have extensively investigated the cloud point extraction of several metal ions with chelating reagents in non-ionic surfactant such Triton X-100 and

---

* Corresponding author. Tel.: +81 29 228 8704; fax: +81 29 228 8403.
E-mail address: oakira@mx.ibaraki.ac.jp (A. Ohashi).

0039-9140/$ – see front matter © 2004 Elsevier B.V. All rights reserved.
doi:10.1016/j.talanta.2004.07.018

poly(oxyethylene)-4-nonylphenyl ether [7–10], and many chelating reagents, until now have been used to extract metal ions for some workers [11–17]. For example, pyridylazo compounds were used for the pre-concentration of Cu(II) [13], Co(II) [14], and Er(III) [15] in the cloud point extraction. Dithizone was employed to the cloud point extraction/pre-concentration on-line flow injection method for Hg(II) determination [16]. 8-Quinolinol was applied for the differentiation and the selective determination of chromium species by using Triton X-114 [17].

In this work, the cloud point extraction behaviors of iron(III) and vanadium(V) using 8-quinolinol derivatives (HA) and Triton X-100 and determination of iron(III) by a graphite furnace atomic absorption spectrometry (GF-AAS) were investigated to get fundamental information concerning the separation of traces metal ions. Furthermore, the photometric determination of iron(III) extracted in the surfactant-rich phase was carried out. The solubility of HA in Triton X-100 aqueous solution was also determined at 25 °C.

2. Experimental

2.1. Reagents

HQ and HMQ (Kanto Chemical Co. Ltd.) were recrystallized twice from ethanol. 5-Alkyloxymethyl-8-quinolinols (HO_nQ, n = 1, 2, 3, 4, 6, 8, 10) and 2-methyl-5-alkyloxymethyl-8-quinolinols (HMO_nQ, n = 1, 2, 3, 4, 6, 8, 10) were prepared by the method previously [18]. Triton X-100 was purchased from Aldrich. Metal stock solutions were prepared by the dilution of AAS metal standard solution (Kanto Chemical Co. Ltd.) with a dilute perchloric acid solution. Water was doubly distilled and further purified with a Milli-Q (Millipore) equipment. An acetate buffer was used to adjust the pH. All other chemicals were of analytical reagent grade and were used without further purification.

2.2. Apparatus

UV–vis spectra were measured with a Jasco V-560 UV/VIS spectrophotometer using the quartz cell with an optical path length of 10 mm. The pH measurements were performed by a Radiometer PHM93 pH meter. A GF-AAS (Shimadzu AA-646) using an autosampler was used to determine iron(III) concentration in aqueous solution and iron(III)–HA complexes in a surfactant-rich phase. The operating condition was as follows: wavelength, 248.3 nm; monochromator band pass, 1 nm; lamp current, 10 mA; sample uptake rate, 10 μl. Vanadium(V) concentration in an aqueous phase was determined by an inductively coupled plasma atomic emission spectrometer (ICP-AES, Nippon Jurrell Ash ICAP-575).

2.3. Solubility measurement of 8-quinolinol derivatives in Triton X-100 solution

Absorption spectra of 4% (v/v) Triton X-100 solution dissolving known amounts of HA were measured to make the calibration curves for HA. The molar absorptivities of HA at maximum wavelength were obtained from these calibration curves.

The solubility of HA in 4% (v/v) Triton X-100 was obtained in the following way. The 4% (v/v) Triton X-100 solution containing excess HA was stirred for 48 h, stayed for 30 min, and centrifuged. The supernatant solution was adequately diluted with 4% (v/v) Triton X-100. Then, absorbance at maximum wavelength of the resulting solution was measured and the solubility of HA was calculated using the molar absorptivity of HA.

2.4. Cloud point extraction procedure

Triton X-100 aqueous solution containing metal ions, acetate buffer, and HA was taken in a test tube with a glass stopper. After staying for 30 min, the solution was heated at 75 °C for 30 min in a thermostated water bath. Then the resulting turbid solution was cooled to about −10 °C for 20 min in a cooling bath and then an aqueous solution was poured off. The aqueous solution was subjected to the analysis of iron(III) by GF-AAS or vanadium(V) by ICP-AES. For measurements of the absorption spectra, the surfactant-rich phase was diluted with water. Then, the resulting solution was supplied to absorption spectral measurements of metal–HA complexes. The volume of the surfactant-rich phase after the phase separation was measured by using a graduated cylinder instead of a test tube, and was obtained from the average of three measurements. The concentration of Triton X-100 and HA were changed in the range of 0.1–8.0% (v/v) and 4.00×10^{-5} to 1.86×10^{-3} M, respectively. The pH was ranged from 1.70 to 5.44.

2.5. Determination of iron(III) in the riverine water reference by GF-AAS

JAC 0031 and JAC 0032 of the riverine water reference were purchased from The Japan Society for Analytical Chemistry. A 10-ml JAC 0031 or 5-ml JAC 0032 was taken in a tube. To the solution, 0.5 ml of 5.00×10^{-2} M HMQ in 4% (v/v) Triton X-100, 2 ml of an acetate buffer solution (pH 5.0), and an adequate amount of 8% (v/v) Triton X-100 were added successively. Then the total volume was made up to 20 ml with water. The solution was kept at 40 °C for 60 min, then heated at 75 °C for 35 min and cooled. After the complete separation of two phases, 0.5 ml of the surfactant-rich phase was taken into 0.5 ml of 0.1 M HCl. The resulting solution was used for GF-AAS analysis.

3. Results and discussion

3.1. Solubility of HA in 4% (v/v) Triton X-100

The solubility of HA in 4% (v/v) Triton X-100 aqueous solution at 25 °C increased in the following order: HO_nQ,

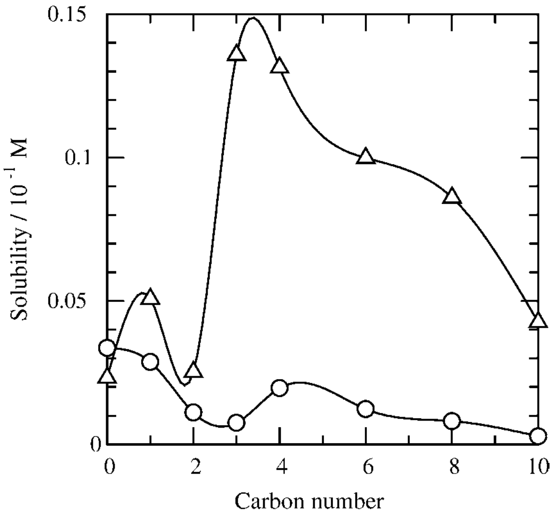

Fig. 1. Effect of an alkyl group on the solubility of 5-alkyloxymethyl-8-quinolinol and 2-methyl-5-alkyloxymethyl-8-quinolinol in 4% (v/v) Triton X-100 at 25 °C. HO$_n$Q (◯), HMO$_n$Q (△).

5-decyloxymethyl-8-quinolinol (HO$_{10}$Q) < 5-propyloxymethyl-8-quinolinol (HO$_3$Q) < 5-octyloxymethyl-8-quinolinol (HO$_8$Q) < 5-ethoxymethyl-8-quinolinol (HO$_2$Q) < 5-hexyloxymethyl-8-quinolinol (HO$_6$Q) < 5-butyloxymethyl-8-quinolinol (HO$_4$Q) < 5-methoxymethyl-8-quinolinol (HO$_1$Q) < 8-quinolinol (HQ); HMO$_n$Q, HMQ < 2-methyl-5-ethoxymethyl-8-quinolinol (HMO$_2$Q) < 2-methyl-5-decyloxymethyl-8-quinolinol (HMO$_{10}$Q) < 2-methyl-5-methoxymethyl-8-quinolinol (HMO$_1$Q) < 2-methyl-5-octyloxymethyl-8-quinolinol (HMO$_8$Q) < 2-methyl-5-hexyloxymethyl-8-quinolinol (HMO$_6$Q) < 2-methyl-5-butyloxymethyl-8-quinolinol (HMO$_4$Q) < 2-methyl-5-propyloxymethyl-8-quinolinol (HMO$_3$Q) (Fig. 1). The solubility of HMO$_n$Q is higher than that of HO$_n$Q. When the number of the ethylene group in HO$_n$Q and HMO$_n$Q molecules is larger than 4 and 3, respectively, the solubility decreased along with an increase in the ethylene group. Though the reason why the solubilities of HO$_3$Q and HMO$_2$Q are relatively small among these HO$_n$Q and HMO$_n$Q molecules has not been clearly explained, the variation in the solubility may be ascribed to a difference in the interaction of alkyl group in the HA molecule with polyethylene group of Triton X-100.

3.2. Extraction behavior of iron(III) with HA

The pH effect on the extraction percentage of iron(III) (2.50×10^{-4} M) with HA (HQ, HMQ, and HMO$_8$Q) (1.25×10^{-3} M) using a 4% (v/v) Triton X-100 aqueous solution was investigated. Iron(III) was completely extracted with HQ, HMQ, and HMO$_8$Q in the surfactant-rich phase at pH 4.85 (Fig. 2). The extraction percentage–pH curve for HMO$_8$Q shifted to a slightly lower pH region than those for HQ and HMQ. Above pH 4.0, more than 95% of iron(III) was extracted with HQ, HMQ, and HMO$_8$Q.

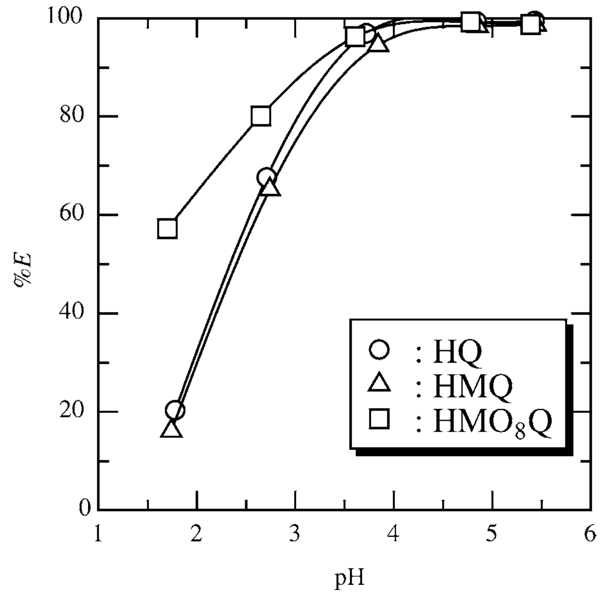

Fig. 2. Effect of pH on the cloud point extraction of iron(III) with HA. Fe(III) 2.50×10^{-4} M, HA 1.25×10^{-3} M, Triton X-100 4% (v/v).

Fig. 3 shows the effect of HA concentration on the cloud point extraction of iron(III) (2.50×10^{-4} M) using a 4% (v/v) Triton X-100 aqueous solution at pH 4.95. The extraction percentage of iron(III) with HA in 4% (v/v) Triton X-100 increased in the following order: HMO$_8$Q < HMQ < HQ. However, the extractability of iron(III) with HMQ was not very different from HQ and HMO$_8$Q. As will be mentioned below, HQ is not adequate for the absorption spectrophotometric determination of iron(III) because of serious interference from vanadium(V). Consequently, HMQ was used for the subsequent cloud extraction experiments.

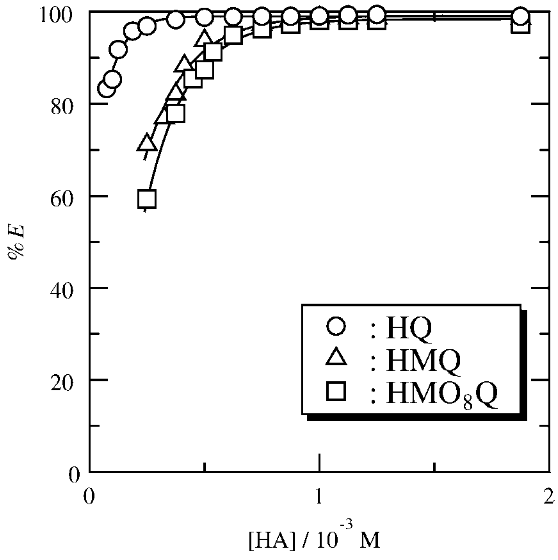

Fig. 3. Effect of HA concentration on the cloud point extraction of iron(III). Fe(III) 2.50×10^{-4} M, Triton X-100 4% (v/v), pH 4.95.

Table 1
Effect of Triton X-100 concentration on the extraction percentage and the concentration factors of iron(III) with HMQ

	Triton X-100 (%, v/v)						
	0.1	0.2	0.5	1.0	2.0	4.0	8.0
V_s (ml)[a]	0.18	0.28	0.39	0.65	1.31	2.61	5.23
%E	92.3	95.2	95.2	95.9	97.1	97.8	85.9
F_{Fe}[b]	102.4	68.0	48.8	29.5	14.8	7.5	3.3

[a] V_s is the volume of the phase-separated surfactant-rich phase. The total volume (the phase-separated surfactant-rich and the aqueous phase) is 20 ml.

[b] F_{Fe} is defined as the iron(III) concentration in the surfactant-rich phase divided by the initial iron(III) concentration.

The effect of the Triton X-100 concentration on the cloud point extraction of iron(III) (2.50×10^{-4} M) with HMQ (1.25×10^{-3} M) at pH 4.85 was investigated. The volume of the surfactant-rich phase after the phase separation (V_s), the extraction percentage (%E) and the concentration factors (F_{Fe}) were determined in a Triton X-100 concentration range of 0.1–8.0% (v/v), where F_{Fe} is defined as the iron(III) concentration in the surfactant-rich phase divided by the initial iron(III) concentration (Table 1). The iron(III) concentration in the surfactant-rich phase was calculated using both values of %E and V_s. The very high value, 102.4, of F_{Fe} was obtained by using 0.1% (v/v) Triton X-100 aqueous solution. More than 95% of iron(III) was extracted with HMQ in the range of 0.2–4% (v/v).

3.3. Extraction behavior of vanadium(V) with HA

The pH effect on the extraction percentage of vanadium(V) (2.00×10^{-5} M) with HA (HQ, HMQ, HO_4Q, and HO_6Q) (2.00×10^{-3} M) using a 4% (v/v) Triton X-100 aqueous solution was investigated. The extractability of vanadium(V) with HA in 4% (v/v) Triton X-100 increased in the following order: HMQ < HQ < HO_4Q < HO_6Q (Fig. 4). Plots of the extraction percentage of vanadium(V) against pH for HO_6Q kept high values (>94.5%) in the experimental pH range. Above pH 2.9, more than 95% of vanadium(V) was extracted with HO_4Q and HO_6Q.

Fig. 5 shows the effect of HA concentration on the cloud point extraction of vanadium(V) (2.00×10^{-5} M) using a 4% (v/v) Triton X-100 aqueous solution at pH 3.40. More than 95% of vanadium(V) was extracted with HO_4Q and HO_6Q above 1.8×10^{-4} and 1.2×10^{-4} M, respectively. The extraction percentage of vanadium(V) with HQ approximately became constant at 77% above 2.4×10^{-4} M.

The effect of the Triton X-100 concentration on the cloud point extraction of vanadium(V) (1.00×10^{-4} M) with HO_6Q (1.00×10^{-3} M) at pH 3.40 was investigated. The extraction percentage and the concentration factors, F_V, were determined in a Triton X-100 concentration range of 0.4–6.0% (v/v) and were listed in Table 2. More than 98% of vanadium(V) was extracted with HO_6Q in all Triton X-100 concentration range.

3.4. Determination of iron(III) in spiked water sample by an absorption spectroscopy

It is worthwhile to apply a spectrophotometric method, which needs neither difficult procedure nor expensive equipment to the determination of metals. The complex of iron(III) with HQ has a broad absorption in the whole visible region, while most of metal–HQ complexes have no absorption around 600 nm [19]. Therefore, HQ has been used as a photometric reagent for iron(III). In this method, vanadium(V) is especially disturbing, because it also gives black vanadium(V)–HQ complex, which has an absorption in the whole visible region.

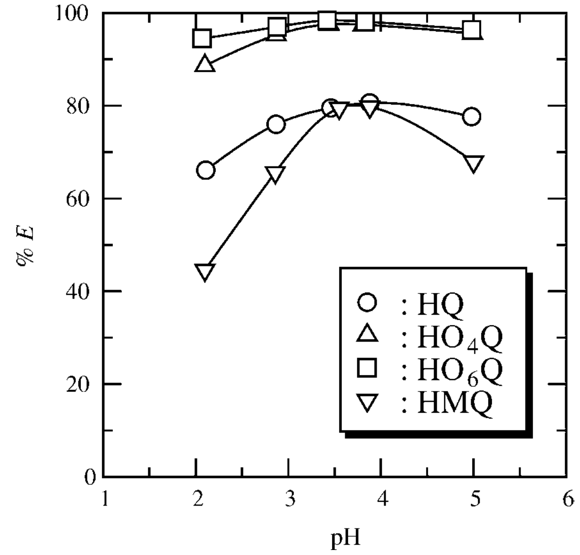

Fig. 4. Effect of pH on the cloud point extraction of vanadium(V) with HA. Vanadium(V) 2.00×10^{-5} M, HA 2.00×10^{-3} M, Triton X-100 4% (v/v).

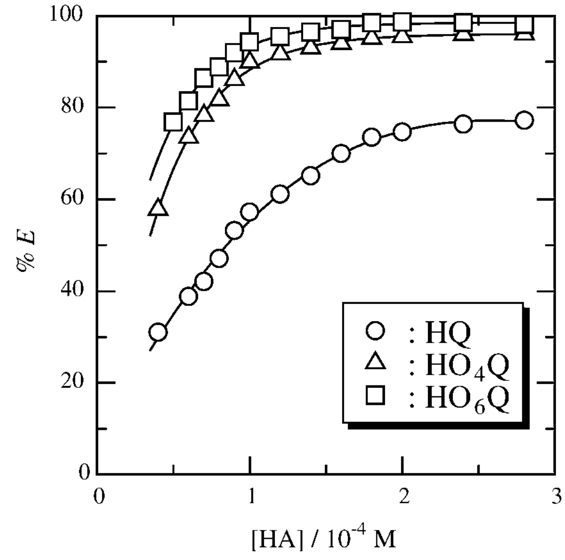

Fig. 5. Effect of HA concentration on the cloud point extraction of vanadium(V). Vanadium(V) 2.00×10^{-5} M, Triton X-100 4% (v/v), pH 3.40.

Table 2
Effect of Triton X-100 concentration on the extraction percentage and the concentration factors of vanadium(V) with HO_6Q

	Triton X-100 (%, v/v)					
	0.4	0.8	1.0	2.0	4.0	6.0
V_s (ml)[a]	0.34	0.55	0.65	1.31	2.61	3.92
%E	98.1	98.4	98.6	98.9	98.6	98.6
F_V[b]	57.7	35.8	30.3	15.1	7.6	5.0

[a] V_s is the volume of the phase-separated surfactant-rich phase. The total volume phase (the phase-separated surfactant-rich and the aqueous phase) is 20 ml.

[b] F_V is defined as the vanadium(V) concentration in the surfactant-rich divided by the initial vanadium(V) concentration.

To test the feasibility of the photometric determination of iron(III) extracted in the surfactant-rich phase, UV–vis absorption spectra of the surfactant-rich phase were measured according to the procedure mentioned above. Absorption spectra of iron(III)- and vanadium(V)–HA complexes extracted in a surfactant-rich phase were obtained as shown in Figs. 6 and 7, respectively. The absorption spectra of iron(III)–HQ, -HMQ, and -HMO_8Q complexes have two absorption maximum wavelength around 470 and 590 nm, whereas, that of vanadium(V)–HMQ complex has no absorption band around 600 nm, although those of vanadium(V)–HQ, -HO_4Q, and -HO_6Q complexes have a broad absorption band around 520 nm. These results suggest that the use of HMQ enables photometric determination of iron(III) in the presence of vanadium(V). Consequently, the determination of iron(III) with HMQ was carried out by an absorption spectroscopy in the cloud point extraction system containing some foreign metal ions.

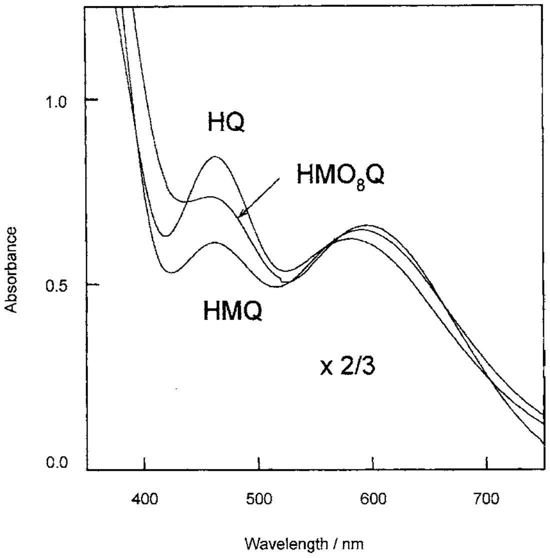

Fig. 6. Absorption spectra of iron(III)–HA complex extracted in surfactant-rich phase. Fe(III) 2.50×10^{-5} M, HA 1.25×10^{-3} M, Triton X-100 4% (v/v), pH 4.94. Surfactant-rich phase was diluted with water two-thirds.

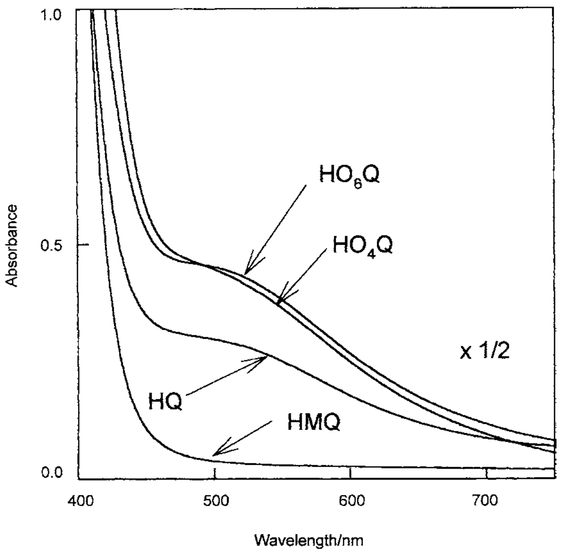

Fig. 7. Absorption spectra of vanadium(V)–HA complex extracted in surfactant-rich phase. Vanadium(V) 2.00×10^{-5} M, HA 2.00×10^{-3} M, Triton X-100 4% (v/v), pH 3.40. Surfactant-rich phase was diluted with water a half.

The effect of foreign metal ions such as vanadium(V), copper(II), nickel(II), cobalt(II), zinc(II), and aluminum(III) on the determination of iron(III) (2.50×10^{-5} M) with HMQ (1.25×10^{-3} M) using a 4% (v/v) Triton X-100 aqueous solution containing foreign metal ions was investigated. In the presence of copper(II), the surfactant-rich phase was tubid. Therefore, the solution was filtered by filter paper before measuring of the absorbance. Based on the calibration curve for iron(III) by the absorbance at 600 nm, 10^{-6} M level of iron(III) could be spectrophotometrically determined. The presence of 2.50×10^{-5} M copper(II) and nickel(II) and 2.50×10^{-4} M vanadium(V), zinc(II), and aluminum(III) ions did not interfere with the determination of iron(III). These results implied the utility of HMQ for the photometric determination of iron(III) in the cloud point extraction system containing some foreign metal ions. The sensitivity of HMQ for iron(III) is lower than those of 1,10-phenanthroline and 4-(2-pyridylazo)resorcinol. However, HMQ can be widely used as the photometric reagent for iron(III), because it is hardly disturbed by the foreign metal ions.

3.5. Application to the determination of iron(III) in the riverine water reference by GF-AAS

The proposed method was applied to the determination of iron(III) in the riverine water reference (JAC 0031 and JAC 0032) by GF-AAS. The cloud point extraction was carried out using 1.25×10^{-3} M HMQ and 1% (v/v) or 4% (v/v) Triton X-100 aqueous solutions. Each sample was analyzed three times. Based on the calibration curve, iron(III) was determined. The analytical results were shown in Table 3. When the Triton X-100 concentration is 1% (v/v), the result showed a good agreement with the certified values within the 2% of the R.S.D. This result shows that the proposed method is

Table 3
Determination of iron(III) in the riverine water reference material by GF-AAS

Reference material	Triton X-100 concentration (%, v/v)	Certified ($\mu g\,l^{-1}$)	Found ($\mu g\,l^{-1}$)	R.S.D. (%)	Recovery (%)
JAC 0031	1.0	6.9 ± 0.5	7.0 ± 0.1	1.1	102
	4.0	6.9 ± 0.5	8.4 ± 0.7	8.0	121
JAC 0032	1.0	57 ± 2	58 ± 1	1.2	101
	4.0	57 ± 2	58 ± 1	1.9	101

sufficiently applied to the determination of 10^{-7} M level of iron(III).

4. Conclusion

The fundamental cloud point extraction behavior of iron(III) and vanadium(V) using 8-quinolinol derivatives and Triton X-100 was investigated. The extraction percentage was influenced by pH, 8-quinolinol derivatives used and their concentrations, and Triton X-100 concentration. The high extraction percentage and concentration factor of iron(III) and vanadium(V) into a surfactant-rich phase could be obtained by the cloud point extraction. A 10^{-6} M level of iron(III) could be spectrophotometrically determined in spiked water sample containing some metal ions by using 1.25×10^{-3} M HMQ and 4% (v/v) Triton X-100 aqueous solution. Moreover, a 10^{-7} M level of iron(III) could be determined in riverine water reference by using 1.25×10^{-3} M HMQ and 1% (v/v) Triton X-100 aqueous solution by GF-AAS.

The results of this work clearly show the utility and validity of the proposed method. The cloud point extraction is an easy, safe, rapid, and inexpensive methodology for separation and pre-concentration of trace metal ions in aqueous phase compared with common liquid–liquid extraction. Since the surfactant-rich phase can be treated directly or after dilution with solvents, such as methanol, the proposed method can be applied to an effective simple pretreatment for several analytical techniques, such as atomic absorption spectrometry, inductively coupled plasma mass spectrometry (ICP-MS), ion-exchange chromatography, and capillary electrophoresis. The detection limit and the selectivity of metal ions can be improved by changing the concentration of the surfactant and chelating agent. The proposed method can be applied to the determination of various trace metal ions in various water samples.

References

[1] H. Watanabe, N. Yamaguchi, Bunseki Kagaku 33 (1984) 211.
[2] M.F. Silva, L. Fernandez, R.A. Olsina, D. Stacchiola, Anal. Chim. Acta 342 (1997) 229.
[3] T. Saitoh, T. Ohyama, T. Sakurai, T. Kaise, K. Takamura, Y. Suzuki, C. Matsubara, Talanta 46 (1998) 541.
[4] R.L. Revia, G.A. Makharadze, Talanta 48 (1999) 409.
[5] Q. Fang, H.W. Yeung, H.W. Leung, C.W. Huie, J. Chromatogr. A 904 (2000) 47.
[6] C.D. Stalikas, Trends Anal. Chem. 21 (2002) 343.
[7] H. Watanabe, H. Tanaka, Talanta 25 (1978) 585.
[8] H. Watanabe, T. Kamidate, S. Kawamorita, K. Haraguchi, M. Miyajima, Anal. Sci. 3 (1987) 433.
[9] T. Saitoh, Y. Kimura, T. Kamidate, H. Watanabe, K. Haraguchi, Anal. Sci. 5 (1989) 577.
[10] H. Watanabe, T. Saitoh, T. Kamidate, K. Haraguchi, Mikrochim. Acta 106 (1992) 83.
[11] C. Wang, D.F. Martin, B.B. Martin, J. Environ. Sci. Health A 34 (1999) 705.
[12] J. Chen, K.C. Teo, Anal. Chim. Acta 450 (2001) 215.
[13] A.N. Tang, D.Q. Jiang, X.P. Yan, Anal. Chim. Acta 507 (2004) 199.
[14] C.C. Nascentes, M.A.Z. Arruda, Talanta 61 (2003) 759.
[15] M.F. Silva, L. Fernandez, R.A. Olsina, D. Stacchiola, Anal. Chim. Acta 342 (1997) 229.
[16] M. Garrido, M.S.D. Nezio, A.G. Lista, M. Palomeque, B.S.F. Band, Anal. Chim. Acta 502 (2004) 173.
[17] E.K. Paleologos, C.D. Stalikas, S.M. Tzouwara-Karayanni, M.I. Karayannis, Anal. Chim. Acta 436 (2001) 49.
[18] K. Ohashi, R. Iwata, S. Mochizuki, H. Imura, K. Hiratani, H. Sugihara, Talanta 43 (1996) 1481.
[19] H. Onishi, Photometric Determination of Traces of Metals, Part IIA, 4th ed., Wiley, New York, 1986, p. 705.