Talanta 105 (2013) 87–92

Contents lists available at SciVerse ScienceDirect

Talanta

journal homepage: www.elsevier.com/locate/talanta

Dispersive liquid–liquid microextraction of trace Hg^{2+} for visual and fluorescence test

Jing Liu [a,b], Dapeng Wu [a,*], Chunfeng Duan [a], Yafeng Guan [a,*]

[a] Department of Instrumentation & Analytical Chemistry, Key Laboratory of Separation Science for Analytical Chemistry of CAS, Dalian Institute of Chemical Physics, Chinese Academy of Sciences, 457 Zhongshan Road, Dalian 116023, China
[b] Graduate School of the Chinese Academy of Sciences, Beijing 100039, China

ARTICLE INFO

Article history:
Received 12 September 2012
Received in revised form
19 November 2012
Accepted 25 November 2012
Available online 1 December 2012

Keywords:
Mercuric ion
Preconcentration
Visual
Fluorescence

ABSTRACT

In this work, trace Hg^{2+} in environmental water samples was first preconcentrated by dispersive liquid–liquid microextraction (DLLME), and then was colorated by one common rhodamine B thiolactone (RBT) probe for visual and fluorescence detection. In this way, a highly sensitive and selective method was developed for field monitoring of Hg^{2+} in environmental waters. The color of RBT test solution could change clearly from colorless to pink around the level of 2 μg L^{-1} Hg^{2+}, which just meets with the maximum residue level of Hg^{2+} in drinking water recommended by U.S. EPA, so the water samples with Hg^{2+} concentration over 2 μg L^{-1} could be selected rapidly by naked eye. Moreover, the quantification of Hg^{2+} could be obtained by fluorescence detection of test solution. The best extraction efficiency of Hg^{2+} by DLLME could be obtained by using 200 mL water sample (pH 5.0, 50 °C), 150 μL extraction solvent (lauric acid, LA) and 3 mL dispersive solvent (methanol). Thus an enrichment factor of ~407 and the limit of fluorescence detection of 0.03 μg L^{-1} could be achieved. The method was also highly selective for Hg^{2+} with tolerance to at least 1000-fold of the foreign metal ions. This method was successfully applied to detect Hg^{2+} in deionized water, tap water and sea water.

1. Introduction

Mercuric ions, widely distributed in water, soil and air [1], poses severe threats to both human health and the environmental bioecology [2] when it is presented in excess concentration. Therefore, it is highly desirable to develop sensitive and selective methods for quantitative detection of Hg^{2+}. For the low-cost and rapid detection of Hg^{2+}, many methods based on gold nanoparticles [3,4] and molecule chemosensors [5,6] have been developed. However, direct determination of Hg^{2+} at trace levels is still limited due to the low concentration and matrix interferences. Therefore, preconcentration of Hg^{2+} is usually required to improve the detection limits [7].

Various preconcetration techniques including solid phase extraction [8,9], liquid phase extraction [10] and cloud point extraction [11,12] had been applied in determination of trace Hg^{2+} in water samples. A new liquid–liquid microextraction technique namely "dispersive liquid–liquid microextraction" was also reported [13] and successfully used for preconcentration of some metal ions [14–18]. DLLME enables the target metal ions transferred from several hundred milliliter water samples into tens or hundreds of microliter extraction solvent in seconds to minutes and has extremely high enrichment capability. Besides, DLLME is also a fast and simple microextraction technique which is suitable for field detection.

After preconcentration, atomic fluorescence spectrometry (AFS) [19], atomic absorption spectrometry (AAS) [20] and inductively coupled plasma–mass spectrometry (ICP–MS) [21] are widely applied detection methods for their high sensitivity and reliability. However, the bulk instruments impeded their wide adoption in field analysis. Portable methods of selective, sensitive and speedy detection of Hg^{2+} in field application are urgently needed and are still under development.

A practicable method to meet the rapid field detection of Hg^{2+} is the visual test by naked eye. Due to the poor detection limits of visual probes based on gold nanoparticles [3,4] and molecule chemosensors [5,6], the combination of an efficient preconcentration procedure and a readily available visual test probe is a suitable route to achieve visual test of trace Hg^{2+} [22–24]. The combination of hollow fiber supported liquid membrane (HFSLM) extraction and AuNPs probe [22] was first reported to enable the visual detection of Hg^{2+} at 2 μg L^{-1}.

In this study, by combining DLLME with the RBT probe [25,26], which is highly selective for Hg^{2+}, we developed a highly sensitive and selective method for field detection of Hg^{2+} in

* Corresponding authors. Tel.: +86 411 84379570; fax: +86 411 84379590.
E-mail addresses: kfguan@mail.dlptt.ln.cn, guan_yafeng@yahoo.com.cn (Y. Guan).

0039-9140/$ - see front matter © 2012 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.talanta.2012.11.061

water samples. In this method, Hg^{2+} was first trapped into the extraction solvent LA by DLLME, and the LA was then mixed with RBT probe solution. Thus, the water samples with Hg^{2+} concentration over 2 μg L^{-1} could be selected rapidly by naked eye and the quantification of Hg^{2+} could be obtained by fluorescence detection of test solution.

2. Experimental

2.1. Chemicals and apparatus

The standard stock solution of heavy metal ions, except for Ag^{+} with $AgNO_3$, was prepared by dissolving the appropriate amount of heavy metal ions chloride salts in 2% (v/v) diluted HNO_3. These reagents are all of analytical reagent, and were obtained from Jingchun Industry Co., Ltd. (Shanghai, China). Standard solutions were prepared by dilution of the stock solutions daily. Acetonitrile and methanol were of HPLC grade (Tedia, Portland, USA). Rhodamine B was obtained from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). LA was purchased from Bodi Chemical Co., Ltd. (Tianjin, China). Deionized water used throughout this work was Wahaha deionized water (Hangzhou, China). The RBT probe was synthesized from rhodamine B and thiourea by two steps [26]. A Perkin Elmer LS55 spectrofluorimeter was used for fluorescence measurements.

2.2. Sample collection and preparation

Tap water collected from our laboratory and seawater from Xinghai Park (Dalian, China) were used as real water samples. The water samples filtered with 0.45 μm cellulose acetate membrane were analyzed within 48 h after sampling. Before extraction, water samples were adjusted to pH 5.0 with 1 M acetate/acetic acid buffer solution.

2.3. Preconcentration of Hg^{2+} by DLLME

For DLLME, 200 mL of water sample was placed in a 250 mL beaker flask and kept at 50 °C. Then 2 mL of 1 M acetate/acetic acid buffer solution of pH 5 was added. A mixed solution of 150 μL LA and 3 mL methanol was rapidly injected into the water sample, and then the mixture was shaken for 5 min. A cloudy solution consisting of very fine LA droplets dispersed into aqueous sample was obtained. After cooling to room temperature, the solidified LA with enriched Hg^{2+} was separated from water sample by filtration and then was used in fluorescence detection.

For visual test, the volume of water samples and extraction solvent was changed to 1000 mL and 250 μL, respectively. The next procedure was same as above.

2.4. Visual and fluorescence detection of Hg^{2+}

For visual test, the solidified LA was mixed with 0.5 mL of 5 mM RBT solution in acetonitrile, and then the solution was kept at 50 °C for 30 s. Then color of the mixed solution was observed by naked eye, and was recorded by a digital camera (Sony, Japan).

For the quantification of Hg^{2+}, the solidified LA with enriched Hg^{2+} was dissolved in 1 mL methanol, and then 2 mL 500 μM RBT probe in acetonitrile was added. The mixed solution was transferred to a 1 cm quartz cuvette for fluorescence measurements.

3. Results and discussion

3.1. Strategy for preconcentration and detection

Fig. 1 outlines the preconcentration and detection procedure employed in this study. In DLLME, LA dispersed in water sample could form fine droplets with carboxyl groups outwards. The Hg^{2+} was trapped onto the surface of the droplets due to the complexing reaction between Hg^{2+} and carboxyl. When the solidified LA with enriched Hg^{2+} was mixed with RBT probe solution, LA droplet was dissolved, and the Hg^{2+} was released

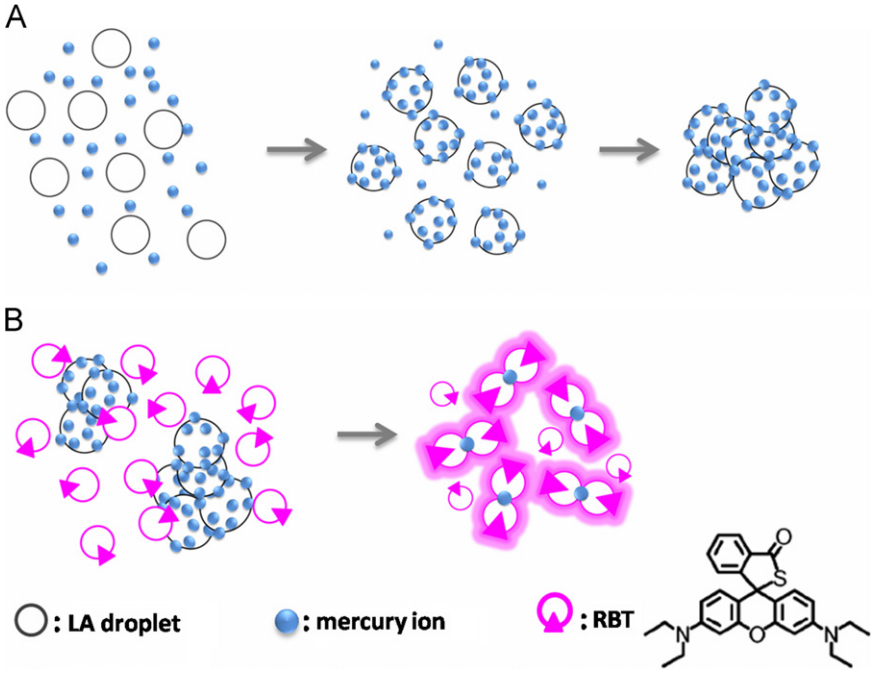

Fig. 1. Scheme for preconcentration (A) and coloration procedure (B) of Hg^{2+} by DLLME–RBT.

from LA. Hg^{2+} ions could induce structural change of RBT probe from colorless and nonfluorescent spirocyclic form to colored and fluorescent open form [25,26]. The reaction is very rapid and specific due to the high thiophilicity of Hg^{2+} and strong nucleophilicity of sulfur [27–29]. In this way, the concentration of Hg^{2+} was detected based on the color change and the fluorescence increase of the RBT probe.

3.2. Optimization of the DLLME conditions

The solution of RBT probe itself displayed colorless and nonfluorescent, but it immediately yielded pink color and strong yellow fluorescence at about 580 nm after mixed with the solidified LA with enriched Hg^{2+}. In this experiment, the increase of fluorescence at 580 nm was selected as a response variable for the optimization of DLLME procedure. Deionized water (200 mL) spiked with 5.0 μg L^{-1} Hg^{2+} was used to study the extraction performance under different conditions. Various factors, such as extraction time, the pH, the volume of extraction agent and dispersive solvent, were optimized for the high efficient preconcentration of Hg^{2+}.

3.2.1. Selection of extraction agent and dispersive solvent

The selection of extraction agent and dispersive solvent is critical for obtaining an efficient DLLME extraction. In this experiment, the extraction reagent should be able to complex with Hg^{2+} effectively and would not be miscible with water. LA was one long chain alkyl carboxylic acid. Its long alkyl chain makes it hardly soluble in water, and its carboxylic acid head group can complex strongly with Hg^{2+}. Thus, LA was selected in this work as the extraction agent. In addition, the melting point of LA is about 46 °C, as the sample solution was cooled down to room temperature, it could solidify quickly, and be collected easily from water sample by filtration. In DLLME, the dispersive solvent should be miscible with both water and LA. Acetone, methanol, ethanol and acetonitrile were investigated in the study. It was found that only methanol could form fine organic droplets with LA in water and provide the best extraction efficiency. Consequently, LA and methanol were used as extraction agent and dispersive solvent in further experiments.

3.2.2. Extraction time

As shown in Fig. 2a, the influence of extraction time on the extraction efficiency was studied over the range from 1 to 20 min. It was found that the extraction processes was rapid, and the extraction efficiency was nearly independent of extraction time after 5 min. It could be attributed to the large interface area between dispersed LA droplets and the aqueous sample phase [13], so the equilibrium of the complexing reaction could be reached quickly after the formation of dispersive cloudy solution. An optimum extraction time was determined as 5 min.

3.2.3. Extraction agent volume

In order to achieve higher Hg^{2+} enrichment factor, the extraction agent volume from 50 to 300 μL was studied. It was found that the fluorescence intensity was rising as the increase of LA volume up to 150 μL, and then to decline (Fig. 2b). The reason was suggested as that with the increase of LA, more Hg^{2+} was extracted from water, but the excess LA will also compete with RBT probe for Hg^{2+} in subsequent coloration reaction. In addition, with the increase of LA, the polarity of test solution decreased and there was a case of blue shift of fluorescence spectra, so the fluorescence intensity at 580 nm decreased. Therefore, 150 μL LA was chosen for 200 mL water sample.

3.2.4. Dispersive solvent volume

The dispersive solvent methanol will help micro droplet formation of LA in water sample, but it can also increase the solubility of LA in water sample especially when it was added in excess. Thus, the dispersive solvent volume had significant effect on the DLLME extraction speed and efficiency. A series of different volumes of methanol from 1.0 to 5.0 mL were studied. According to Fig. 2c, 3.0 mL methanol could lead to the best extraction

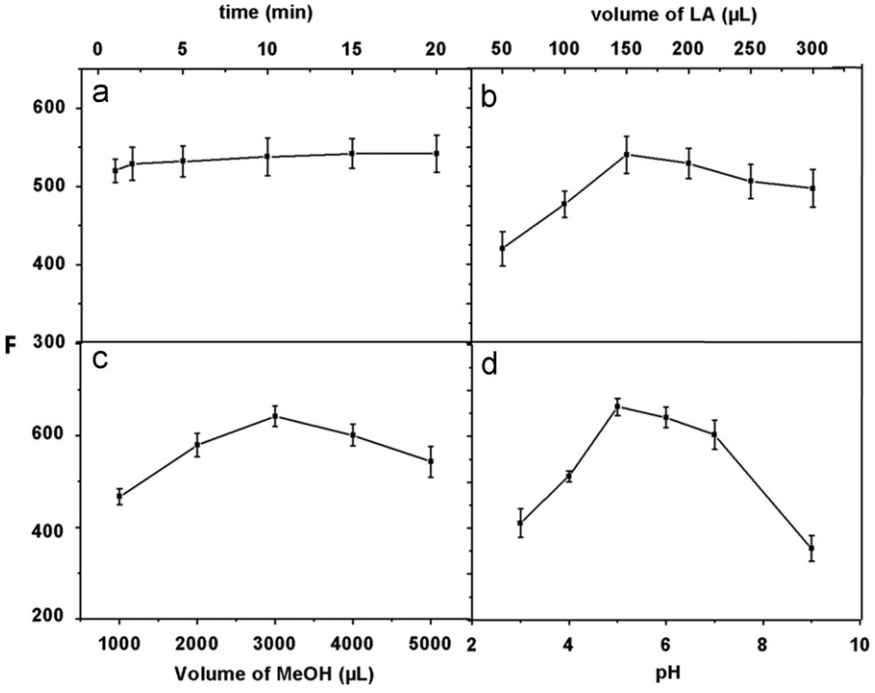

Fig. 2. Effect of (a) extraction time, (b) volume of extraction agent, (c) volume of dispersive solvent and (d) pH value of water samples in DLLME on the extraction efficiency of Hg^{2+} represented by fluorescence increase at 580 nm. Ex slit: 5 nm; and Em slit: 5 nm.

efficiency, and was chosen as the optimal dispersive solvent volume.

3.2.5. Sample pH

pH plays a unique role in the complexing reaction. It was revealed that the best extraction efficiency was obtained at a pH about 5 (Fig. 2d). In acidic medium, LA tends to be fully acidified and shows low affinity to Hg^{2+}. On the contrary, at a higher pH than 5, metal hydroxide species, such as soluble $M(OH)^{+}$ and/or insoluble precipitate of $M(OH)_n$, were formed, which could not be extracted by LA. Hence, pH of 5.0 was selected, and the rest of the experiments were carried out in the presence of a diluted 0.01 M acetate/acetic acid buffer solution of pH 5.

3.3. Optimization of fluorescent and chromogenic reaction

Under the above optimized DLLME conditions, other parameters that would affect the reaction of Hg^{2+} and RBT probe were further studied to improve the DLLME–RBT method.

3.3.1. Test solution volume

In fluorescence detection, as the solidified LA could not dissolve directly in 3 mL acetonitrile, the solidified LA was first dissolved into 1 mL methanol, and then 2 mL of RBT probe in acetonitrile was added. The total test solution of 3 mL was transferred to quartz cuvette for the fluorescence detection. But for a sharp and clear color change in visual detection, the test solution should be as small as possible, so the solidified LA was heated at 50 °C to a liquid state instead of dissolved in 1 mL methanol.

3.3.2. Reaction time

The reaction time between RBT probe and Hg^{2+} was defined as the time interval between the mix of solidified LA with RBT probe solution and the fluorescence detection. It was found that the time required to reach the steady state was very fast, so the mixture was detected immediately after RBT probe solution was added.

3.3.3. RBT probe concentration

For visual detection, it was found that color of test solution was changed to pink obviously when 0.5 mL of 5.0 mM RBT probe in acetonitrile was added. So 0.5 mL of 5 mM RBT probe in acetonitrile was selected to mix with solidified LA for visual detection. In fluorescence detection, the sensitivity depends strongly on the fluorescence reagent. Results shown in Fig. 3 revealed that the fluorescence signal kept stable when the concentration of RBT was higher than 300 μM. Excessive RBT probe could promote the release of Hg^{2+} from LA and the reaction between Hg^{2+} and RBT, so RBT of 500 μM was adopted in the subsequent studies for fluorescence detection. In addition, excessive RBT probe could also ensure the applicability of this method for water samples with Hg^{2+} over 5.0 μg L^{-1}.

3.4. Analytical performance

Selectivity and sensitivity of the proposed DLLME–RBT method were evaluated by using standard solution based on fluorescence increase and color change of test solution.

3.4.1. Selectivity

In environmental water, most foreign metal ions have much higher concentration than Hg^{2+}. To assess the specificity of this method, Hg^{2+} at the concentration of 10 μg L^{-1} (50 nM) and other various ions (Ag^{+}, Pb^{2+}, Cd^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Fe^{3+}, Mg^{2+}, Ba^{2+}, Ca^{2+}, and Al^{3+}) with a concentration of 50 μM were examined in parallel under the same condition. As shown in Fig. 4, the test solution of foreign metal ions were still colorless, whereas only the Hg^{2+} sample displayed pink. To quantify the Hg^{2+} specificity of the method, fluorescence measurements for Hg^{2+} and the foreign metal ions were also performed. It was found that no significant spectral changes occurred for all these foreign metal ions (Fig. 5), so the proposed DLLME–RBT method responded selectively toward Hg^{2+} by a factor of at least 1000-fold to the foreign metal ions.

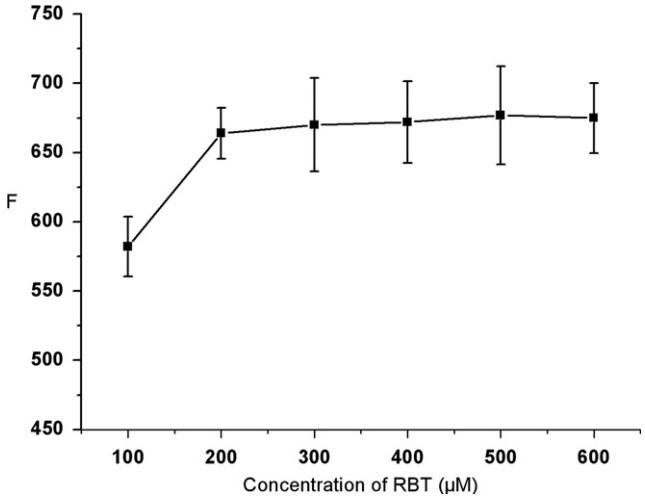

Fig. 3. Effect of the concentration of RBT probe on the detection of Hg^{2+} represented by fluorescence increase at 580 nm. Ex slit: 5 nm; and Em slit: 5 nm.

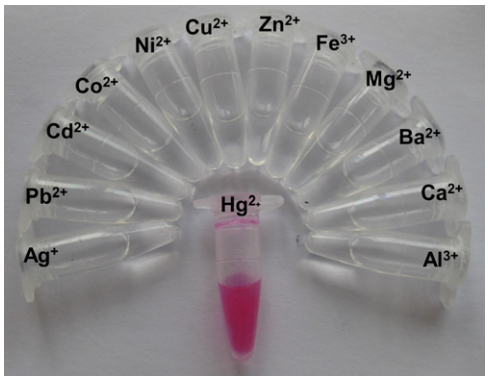

Fig. 4. Photographs of DLLME–RBT test solution for 10.0 μg L^{-1} Hg^{2+} and other foreign metal ions at concentration of 50 μM. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)

3.4.2. Sensitivity

In order to achieve a visual test of Hg^{2+} at concentration of 2.0 μg L^{-1}, the conditions of the DLLME–RBT procedure had to be adjusted correspondingly, as the test solution of the following chromogenic assay remained colorless for 200 mL water samples containing 2.0 μg L^{-1} Hg^{2+}. It was found that the test solution became clearly pink when the volume of water sample and LA were 1000 mL and 250 μL, respectively. As shown in Fig. 6a, the test solution became slightly pink for water sample containing 1.0 μg L^{-1} Hg^{2+}, and showed colorless to naked eyes for 0.5 μg L^{-1} Hg^{2+}. For the high enrichment capability of DLLME, this method allows detection of Hg^{2+} in water sample around 2.0 μg L^{-1} with the naked eye. Water samples with concentration of Hg^{2+} over 2.0 μg L^{-1} could be selected quickly by the color

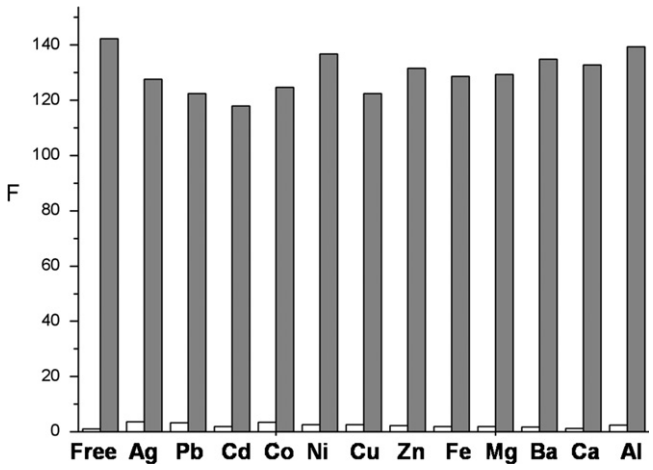

Fig. 5. Fluorescence increase at 580 nm to various cations. The white bars represent the fluorescence increase of 50 μM of Pb^{2+}, Cd^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Fe^{3+}, Mg^{2+}, Al^{3+}, Ca^{2+} and Ba^{2+}. The gray bars represent the fluorescence increase upon the subsequent addition of 10.0 μg L^{-1} of Hg^{2+}. Ex slit: 5 nm; and Em slit: 2.5 nm.

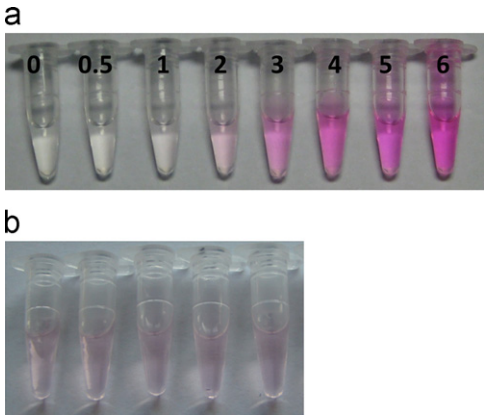

Fig. 6. Photographs of DLLME–RBT test solution for different concentration of Hg^{2+} from 0 to 6.0 μg L^{-1} (a), and photographs of DLLME–RBT test solution for 5 water samples with concentration of Hg^{2+} at 2.0 μg L^{-1} (b). (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)

Table 1
Analytical characteristics for determination of Hg^{2+} by DLLME–RBT.

Analytical characteristics	DLLME–RBT
Linear equation	$Y=-39.7+139X$
Linear range (μg L^{-1})	0.5–6
Correlation coefficient (R)	0.996
Repeatability[a] (*RSD*, %) ($n=5$)	3.1
Limit of detection ($S/N=3$, μg L^{-1})	0.03
Limit of quantification ($S/N>10$, μg L^{-1})	0.11

[a] The solution containing 2. 0 μg L^{-1} of Hg^{2+} was analyzed.

change of test solution, and the quantification could be achieved by fluorescence detection.

After DLLME–RBT, the fluorescence of test solution showed significant increase. In order to evaluate the DLLME–RBT method, limits of detection (LOD) and quantification (LOQ), repeatability, working range and linearity were assessed under the optimized conditions. Results were listed in Table 1. The limit of detection (LOD) at S/N of 3 and the limits of quantification (LOD) at S/N of 10 were 0.03 μg L^{-1} and 0.11 μg L^{-1}, respectively. The method showed good linearity over the calibration range (0.5–6 μg L^{-1}) and the correlation coefficient (r) was 0.996 for Hg^{2+}. This demonstrated that DLLME–RBT is suitable for quantitative analysis of Hg^{2+} at the parts per billion levels by fluorometry.

3.4.3. Repeatability

The repeatability of this method was evaluated by assaying five standard solutions with 2.0 μg L^{-1} Hg^{2+}. For all the assays, as shown in Fig. 6b, a clearly pink color of test solution was observed by the naked eye. In fluorescence detection, a relative standard deviation (*RSD*) of 3.1% (Table 1) illustrated the good repeatability achieved by the procedure.

3.5. Method validation and application

The proposed method was applied to the determination of Wahaha deionized water and two real water samples including tap water and ocean water. In order to validate the ability of visual test, 1000 mL of each water sample spiked with 0, 1.0, 1.5, 2.0 and 3.0 μg L^{-1} Hg^{2+} was measured. Photographic images of Fig. 7 indicated that the color of test solution of all real water samples changed obviously to pink at spiked with 2.0 μg L^{-1} Hg^{2+}, and became slightly pink at 1.0 μg L^{-1}. The results were in good agreement with that obtained by standard solution.

In order to achieve quantitative analysis of Hg^{2+}, water samples of 200 mL were applied for fluorescence detection. As shown in Table 2, despite the very low detection limit of this method by fluorometry, no significant fluorescence increase could be detected for real water samples. The relative recoveries were 96.0% and 93.5% for the spiked deionized water and tap

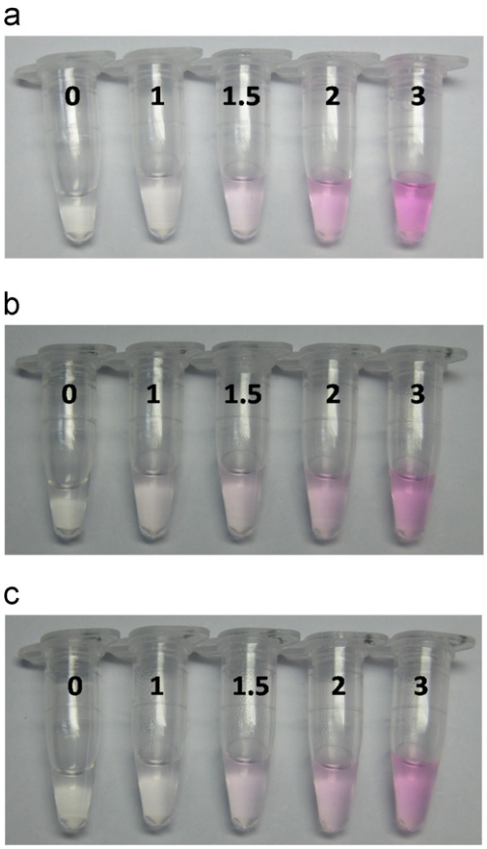

Fig. 7. Photographs of test solution for (A) Wahaha deionized water, (B) tap water and (C) sea water. Each water sample was spiked with 0, 1.0, 1.5, 2.0, 3.0 μg L^{-1} Hg^{2+}. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)

Table 2
Analytical results of Hg^{2+} in water samples and of the spike-recovery tests.

Water sample	Spiked ($\mu g\,L^{-1}$)	Measured ($\mu g\,L^{-1}$, mean ± S.D., $n=5$) and average recovery (%, in the parallel)
Deionized water	0	ND[a]
	2.0	1.92 ± 0.06 (96.0)
Tap water	0	ND
	2.0	1.87 ± 0.08 (93.5)
Sea water	0	ND
	2.0	1.57 ± 0.16 (78.5)

[a] Not detected.

water respectively, but it was only 78.5% for sea water sample. The reason may be that the large amount of salt in sea water could prevent the formation of fine organic droplets during DLLME, which would induce the decrease of extraction efficiency.

4. Conclusion

By the combination of DLLME concentration and RBT probe, a DLLME–RBT method was developed, which allowed the visual and fluorescence detection of Hg^{2+} at the EPA-desired 2.0 $\mu g\,L^{-1}$ level. Because of the high enrichment capability of DLLME and the selective response to Hg^{2+} by using RBT probe, the proposed method showed excellent sensitivity and selectivity towards Hg^{2+}. The recognition of Hg^{2+} at 2.0 $\mu g\,L^{-1}$ level gave sharp color change from colorless to pink, which was clearly visible to the naked eye. The precise Hg^{2+} quantification could be achieved by fluorescence increase of RBT probe. This rapid, selective and sensitive method holds great potential for the field detection of Hg^{2+} in real water samples.

Acknowledgments

The financial support of this research by National Nature Science Foundation of China (Nos. 21027001, 21005080, 21021004) and by China 863 project (No. 2011AA060602) is gratefully acknowledged.

References

[1] J.R. Miller, J. Rowland, P.J. Lechler, M. Desilets, L.C. Hsu, Water Air Soil Pollut. 86 (1996) 373–388.
[2] Q.R. Wang, D. Kim, D.D. Dionysiou, G.A. Sorial, D. Timberlake, Environ. Pollut. 131 (2004) 323–336.
[3] H.N. Kim, W.X. Ren, J.S. Kim, J. Yoon, Chem. Soc. Rev. 41 (2012) 3210–3244.
[4] Y.W. Lin, C.C. Huang, H.T. Chang, Analyst 136 (2011) 863–871.
[5] H.N. Kim, M.H. Lee, H.J. Kim, J.S. Kim, J. Yoon, Chem. Soc. Rev. 37 (2008) 1465–1472.
[6] E.M. Nolan, S.J. Lippard, Chem. Rev. 108 (2008) 3443–3480.
[7] N.J.K. Simpson, Solid Phase Extraction; Principles, Techniques and Application, Marcel Dekker, USA, 2000.
[8] M.E. Mahmoud, M.M. Osman, M.E. Amer, Anal. Chim. Acta 415 (2000) 33–40.
[9] Y.G. Yin, M. Chen, J.F. Peng, J.F. Liu, G.B. Jiang, Talanta 81 (2010) 1788–1792.
[10] I. Lopez-Garcia, R.E. Rivas, M. Hernandez-Cordoba, Anal. Bioanal. Chem. 396 (2010) 3097–3102.
[11] I.M. Dittert, T.A. Maranhao, D.L.G. Borges, M.A. Vieira, B. Welz, A.J. Curtius, Talanta 72 (2007) 1786–1790.
[12] M. Garrido, M.S. Di Nezio, A.G. Lista, M. Palomeque, B.S.F. Band, Anal. Chim. Acta 502 (2004) 173–177.
[13] M. Rezaee, Y. Assadi, M.R.M. Hosseinia, E. Aghaee, F. Ahmadi, S. Berijani, J. Chromatogr. A 1116 (2006) 1–9.
[14] Y. Yamini, M. Rezaee, A. Khanchi, M. Faraji, A. Saleh, J. Chromatogr. A 1217 (2010) 2358–2364.
[15] P. Liang, L. Zhang, E. Zhao, Talanta 82 (2010) 993–996.
[16] Q. Zhou, N. Zhao, G. Xie, J. Hazard. Mater. 189 (2011) 48–53.
[17] M. Mirzaei, M. Behzadi, N.M. Abadi, A. Beizaei, J. Hazard. Mater. 186 (2011) 1739–1743.
[18] V.A. Lemos, L.O. dos Santos, S. Silva, E.V. dos Santos Vieira, J. AOAC Int. 95 (2012) 227–231.
[19] Y.G. Yin, J.F. Liu, B. He, E.L. Gao, G.B. Jiang, J. Anal. Atom. Spectrom. 22 (2007) 822–826.
[20] O. Alp, N. Ertas, J. Anal. Atom. Spectrom. 24 (2009) 93–96.
[21] M. Wang, W.Y. Feng, H.J. Wang, Y. Zhang, J. Li, B. Li, Y.L. Zhao, Z.F. Chai, J. Anal. Atom. Spectrom. 23 (2008) 1112–1116.
[22] Z.Q. Tan, J.F. Liu, Anal. Chem. 82 (2010) 4222–4228.
[23] Z.Q. Tan, J.F. Liu, R. Liu, Y.G. Yin, G.B. Jiang, Chem. Commun. (2009) 7030–7032.
[24] A. Gotzl, W. Riepe, Talanta 54 (2001) 821–827.
[25] X.Q. Zhan, Z.H. Qian, H. Zheng, B.Y. Su, Z. Lan, J.G. Xu, Chem. Commun. (2008) 1859–1861.
[26] W. Shi, H. Ma, Chem. Commun. (2008) 1856–1858.
[27] R.A. Batey, D.A. Powell, Org. Lett. 2 (2000) 3237–3240.
[28] K.C. Song, J.S. Kim, S.M. Park, K.C. Chung, S. Ahn, S.K. Chang, Org. Lett. 8 (2006) 3413–3416.
[29] B. Liu, H. Tian, Chem. Commun. (2005) 3156–3158.