Scientific paper

Spectrophotometric Determination of Fe^{3+} and Pb^{2+} in Real Samples after Micelle-mediated Extraction

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Abstract

A cloud-point extraction process using micelle of the cationic surfactant CTAB to extract $Fe^{3+}$and Pb^{2+} from aqueous solutions was investigated. The method is based on the color reaction of $Fe^{3+}$and Pb^{2+} with bromopyrogallol red at pH 5.0 and micelle-mediated extraction of the complex. The optimal extraction and reaction conditions (e.g., surfactant concentration, reagent concentration and effect of time) were studied and the analytical characteristics of the method (e.g., limit of detection, linear range, preconcentration and improvement factors) were obtained. Linearity was obeyed in the range of 0.05–43.00 ng mL^{-1} of Fe^{3+} ion and 0.10–40.00 ng mL^{-1} of Pb^{2+} ion and the detection limit of the method was 0.020 and 0.040 ng mL^{-1} for Fe^{3+} and Pb^{2+}, respectively. The interference effect of some anions and cations was also tested. The method was applied to the determination of Fe^{3+} and Pb^{2+} in vegetable (lettuce, spinach, cabbage, parsley, and dill), tea, rice, human hair, liver, and chicken and water samples.

Keywords: Fe^{3+}; Pb^{2+}; Bromopyrogallol red; Foodstuff; Human hair, Spectrophotometry.

1. Introduction

Lead is one of the most toxic metals[1,2] and has accumulative effect. It is called environmental priority pollutants.[3] It also increases blood pressure and causes weakness in fingers, wrists and ankles. Moreover, exposure to high level of lead can severely damage kidneys and brain.[4] Lead causes a decrease in the rate of globulin and hem synthesis. Symptoms of lead poisoning include renal insufficiency, colic, constipation and other gastrointestinal effects. It also affects the reproductive system, resulting in sterility, abortions, still birth and neonatal deaths.[5] Plants can absorb lead from the soil, fertilizers and air accumulating in the tissue, thus it can reach the human chain feeding.[6] Lead hidden in foods is one of the main sources of lead absorbed in human body, so the determination of lead in foods is becoming increasingly important.[7] Iron is widely distributed in nature[8] and is one of the most important elements in geochemical, environmental and biological systems.[9] Lack of this element in the daily diet may result in the development of serious diseases such as iron deficiency anemia.[10] However, excess uptake of iron through water pollution results in acute and/or chronic poisoning.[11] Iron is present in very low concentrations in the oceans, despite its enhanced abundance in the earth's crust, and is a vital constituent of plant life. The element plays an important role in plant metabolism where it is essential for photosynthetic and respiratory electron transport, nitrate reduction, chlorophyll synthesis and detoxification of reactive oxygen species.[12] Iron is necessary for hemoglobin synthesis and oxidative processes of living tissues, as it exists at the active site of molecules responsible for oxygen transport, and provides a fundamental structure of myoglobin, hemenzymes, and many co-factors involved in enzyme activities.[13]

It is present as Fe^{3+} and Fe^{2+} states in natural waters, but it is usually present in the Fe^{3+} state.[14] This diversity of biological functioning and sources makes it a prime necessity for an accurate determination of iron and lead at trace levels in various matrices. Different analytical techniques such as x-ray spectroscopy (XRS),[15] flame atomic absorption spectrometry (FAAS),[16,17] electrothermal atomic absorption spectrometry (ET-AAS),[18] inductively coupled plasma mass spectrometry (ICP-MS),[19] and voltammetric methods[9,13,20] may be used for the trace determination of iron and lead in complex materials, but they use sophisticated instruments. That, their day to day maintenance cost is high and they are not free from various types of interferences.

Spectrophotometric methods are most commonly used method for the determination of iron and lead, especially in developing countries. A number of reagents including Tiron,[21] norfloxacin,[22] 1,10-phenanthroline,[23] BTAS,[24] thiocyanate,[25] chlortetracycline,[15] dithizone,[6,26] dibromo-*p*-methyl-methylsulfonazo and Camphor-3-thioxo-2-oxime (HCTO)[27] have been proposed for the determination of these elements. However, due to the presence of iron and lead in environmental and biological samples at low levels, its separation from other elements presents and also the use of a preconcentration is usually necessary.

Cloud-point extraction (CPE), based on the clouding phenomena of surfactants, offers many advantages over traditional liquid-liquid extraction.[28] For charged micelles, the phenomenon rarely occurs, presumably because electrostatic repulsion prevents phase separation in most cases. In the presence of salt, long-tailed cationic surfactants can self-assemble in aqueous solution into long, flexible wormlike micelles, thus rendering the solution viscoelastic.[29] High concentrations of salt cause cationic surfactant solutions to separate into immiscible surfactant-rich and surfactant-poor phases.[30]

Recently, the cloud-point extraction was used for preconcentration of trace quantities of some cations prior to their determination by spectrophotometric method.[31–36] This paper proposes a method to preconcentration and determination of iron and lead by spectrophotometry based on cloud-point extraction (CPE) of the complex of Fe^{3+} and Pb^{2+} with bromopyrogallol red in surfactant media.

2. Experimental

2. 1. Apparatus

A Perkin-Elmer Lambda-45 UV/Vis spectrophotometer was used for recording absorbance spectra. Absorption measurements at fixed wavelength were performed using a Shimadzu UV-mini- 1240V spectrophotometer with 1-cm quartz cell (0.5 mL). A Metrohm pH meter (model 713) with a combined glass electrode was used for pH measurements. A water bath with good temperature control and a centrifuge with 10 mL calibrated centrifuge tubes (Superior, Germany) were used to accelerate the phase separation process.

2. 2. Reagents

The surfactant, cetyltrimethylammounium bromide (CTAB) obtained from Sigma Company (St. Loius, MO, USA) was used without further purification. Stock solutions of Fe^{3+} and Pb^{2+} at a concentration of 1000 μg mL^{-1} was prepared by dissolving appropriate amounts of $FeCl_3 6H_2O$ and $Pb(NO_3)_2$ salts purchased from Merck Company (Darmstadt, Germany) in doubly distilled water. Working standard solutions were obtained by appropriate dilution of the stock solutions. A solution of 1.0×10^{-4} mol L^{-1} of bromopyrogallol red (BPR), (obtained from Merck) was prepared by dissolving appropriate amounts of this reagent in doubly distilled water. A pH 5.0 acetate buffer solution (0.1 mol L^{-1}) was prepared from acetic acid and sodium acetate.[37] N,N-Dimethyl formamide (DMF) solvent and potassium iodide salt were purchased from Merck Company.

2. 3. Procedure

An aliquot of Fe^{3+} and Pb^{2+} standard solutions (0.5–430.0 ng of Fe^{3+} ion and 1.0–400.0 ng of Pb^{2+} ion) was transferred in to a 10 mL centrifuge tube, 0.3 mL of 7.2×10^{-5} mol L^{-1} BPR solution and 3.0 mL buffer solution were added to it. This was followed by the addition of 1.0 mL of 3.0×10^{-3} mol L^{-1} surfactant CTAB solution and 1.0 mL of 0.2 mol L^{-1} of KI solution. The solution was taken up to the mark with doubly distilled water and allowed to stand for 10 min in room temperature. Separation of the aqueous and surfactant-rich phase was accomplished by centrifugation for 15 min at 3800 rpm. Then, the aqueous phase could be separated by inverting the tube. The surfactant-rich phase of this procedure was dissolved and diluted to 0.5 mL with the DMF and transferred into a 0.5 mL quartz cell. The absorbance of the Fe^{3+} solution was measured at 630 nm and Pb^{2+} solution was measured at 576 nm. The blank solution was submitted to the same procedure and its absorbance was measured at 630 and 576 nm, for Fe^{3+} and Pb^{2+}, respectively.

2. 4. Analysis of Real Samples

For water analysis, two water samples given from Ganjnameh River (Hamedan city) and tap water (Hamedan city) were selected for analysis. Each sample was filtered using filter paper (Whatman No.1). Then, 1.0 mL of nitric acid was added into 100 mL of the sample and heated up to dryness to destroy organic compounds. The residue was dissolved in 50 mL of water in a 50 mL volumetric flask. Then, suitable aliquots were taken and analyzed for Fe^{3+} using the proposed procedure.

In order to digest of various food and hair samples, three types of digestion procedures were applied. The blank solutions were prepared together during the analysis of each sample. The analyses of sample and blank solution were performed in five replicates. The dissolution procedures were adopted from various literatures[38–40] (see below) and are as follows.

For the determination of Fe^{3+} and Pb^{2+} in vegetable samples, about 1.0 g of the dried sample placed in 100 mL beaker and 5 mL of concentrated HNO_3 (65%, w/w) was added. The mixture was heated on a hotplate to near dryness. The residue was dissolved in 20 mL of distilled water. The solution was filtered using filter paper (Whatman No. 1) and the filtration was collected into a 50 mL volu-

metric flask and diluted to the mark with distilled water. Suitable aliquots were taken and analysed for Fe^{3+} and Pb^{2+} using the proposed procedures. The amounts of Fe^{3+} and/ or Pb^{2+} in the sample solution were deduced from the calibration curve.

Hair samples were washed with a 0.1 % detergent solution that contained no detectable zinc, 95 % ethanol and ethyl ether. About 0.5 g of the dried sample placed in 100 mL beaker and 5 mL of concentrated HNO_3 was added. The mixture was heated to near dryness. The residue was dissolved with distilled water. The solution was filtered and the filtration was collected into a 50 mL volumetric flask and diluted to the mark with distilled water. Suitable aliquots were taken and analyzed for Fe using the proposed procedures. The amount of Fe^{3+} in the sample solution was deduced from the calibration curve.

About 10.0 g of the fresh liver and chicken sample was first ashed for 6 h at 500 °C in a crucible. After cooling, the ash was carefully moistened with 5 mL of 1:1 nitric acid and the mixture was heated on a hotplate to near dryness. The residue was dissolved in 20 mL of distilled water. The solution was filtered using filter paper (Whatman No. 1) and the filtration was collected into a 50 mL volumetric flask and diluted to the mark with distilled water. Suitable aliquots were taken and analysed for Fe^{3+} and Pb^{2+} using the proposed procedures. The amounts of Fe^{3+} and/or Pb^{2+} in the sample solution were deduced from the calibration curve.

3. Results and Discussion

Bromopyrogallol red is often used as a chromogenic reagent for the determination of a large number of metals, particularly Fe^{3+} and Pb^{2+}.[41] The addition of surfactant-active substances improves the selectivity and sensitivity of the metal determinations due to the batho and hyperchromic effects that can be observed. The literature results for complexation of Fe with BPR in the presence of CTAB showed the component ratio 1:3:6 (Fe: BPR: CTAB).[42] The solution became turbid by addition of the iodide ion. Therefore, the ternary complex of Fe(III)–BPR–CTAB and Pb(II)–BPR–CTAB can be extracted by CPE method. Complexes of Fe^{3+} and Pb^{2+} with BPR in the presence of CTAB in aqueous media and surfactant rich media have maximum absorbance at 630 and 576 nm, respectively. (Figs. 1 and 2). After separation of surfactant-rich phase, the absorbance was measured at 630 and 576 nm, for Fe^{3+} and Pb^{2+}, respectively, against a reagent blank as the reference.

3. 1. Optimization of the System

To take full advantage of the procedure, the reagent concentrations and reaction conditions must be optimized. Various experimental parameters were studied in order to obtain optimized system. These parameters were optimized by setting all parameters to be constant and optimizing one each time.

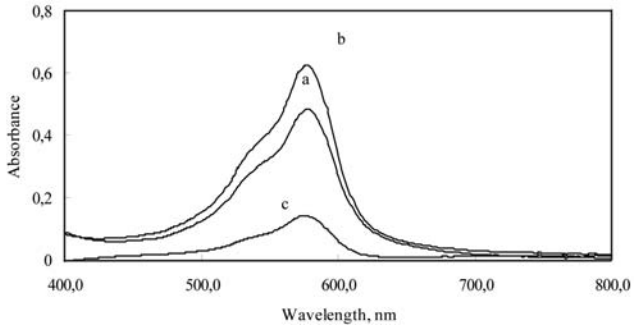

Fig. 1. Absorption spectra for BPR in the presence of CTAB (a) and its complex with Pb^{2+} after extraction in surfactant-rich phase (b); and difference between them (c); conditions: BPR, 2.5×10^{-6} mol L^{-1}; Pb^{2+}, 30 ng mL^{-1}; KI, 0.02 mol L^{-1}; CTAB concentration, 3.0×10^{-4} mol L^{-1}; pH, 5.0

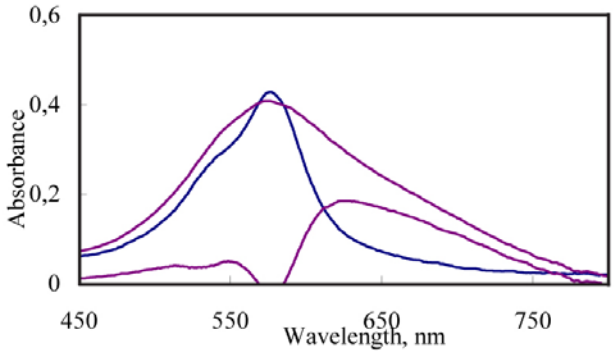

Fig. 2. Absorption spectra for BPR in the presence of CTAB (a) and its complex with Fe^{3+} after extraction in surfactant-rich phase (b); and difference between them (c); conditions: BPR, 3.9×10^{-6} mol L^{-1}; Fe^{3+}, 30 ng mL^{-1}; KI, 0.02 mol L^{-1}; CTAB concentration, 3.0×10^{-4} mol L^{-1}; pH, 5.0.

The effect of pH on the absorbance at a constant concentration of each complex in surfactant-rich phase was investigated in the range 1.0–7.0. The absorbance of the Fe^{3+} system at 630 nm and Pb^{2+}system at 576 nm in surfactant-rich phase was studied against the reagent blank. The results showed that the pH of 5.0 gives the highest sensitivity for determination of Fe^{3+} and Pb^{2+} ions. Therefore, pH 5.0 was selected as optimal (Fig 3).

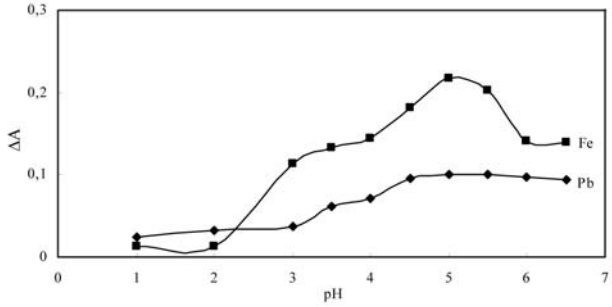

Fig. 3. Effect of pH on the extraction and determination of Fe^{3+} and Pb^{2+} ions

Effect of BPR concentration on the extraction and determination of Fe^{3+} and Pb^{2+} ions was investigated in the range of (0.5 – 4.5) × 10^{-6} mol L^{-1}. The sensitivity of the method increased by increasing BPR concentration up to 3.9 × 10^{-6} mol L^{-1} and 2.5 × 10^{-6} mol L^{-1} for Fe^{3+} and Pb^{2+} ions, respectively. According to Fig. 4 absorbance of complexes at their λ_{max} decreased at higher concentrations. It was expected that increasing BPR causes an increase in the absorbance of complexes, because increasing in BPR concentration can cause an increase in concentration of the complexes. At concentrations higher than 3.9 × 10^{-6} mol L^{-1} for Fe^{3+} and 2.5 × 10^{-6} mol L^{-1} for Pb^{2+}, the concentration of uncomplexed BPR in surfactant-rich phase increased significantly. Therefore, much probably decrease in the net absorbance of complexes at concentrations higher than of BPR is due to this fact that the free BPR competes with the complexes in extraction to surfactant-rich phase. A concentration of 3.9 × 10^{-6} mol L^{-1} and 2.9 × 10^{-6} mol L^{-1} of BPR was selected as the optimum for Fe^{3+} and Pb^{2+} systems, respectively.

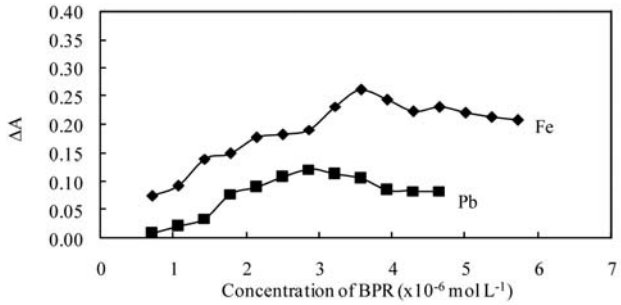

Fig. 4. Effect of concentration of BPR on the extraction and determination of Fe^{3+} and Pb^{2+} ions

Figure 5 shows effect of CTAB concentration in the range of (0.5–4.0) × 10^{-4} mol L^{-1} on the extraction and determination of Fe^{3+} and Pb^{2+} ions. The amount of the absorbance for samples increased by increasing CTAB concentration up to 3.0 × 10^{-4} mol L^{-1} and decreased at higher concentrations. The blank signal also increased by increasing CTAB concentration. This is due to more extraction of BPR by increasing CTAB concentration, but the difference between the sample and blank signals (ΔA) increased by increasing CTAB concentration up to 3.0 × 10^{-4} mol L^{-1} and decreased at higher concentrations. Therefore, 3.0 × 10^{-4} mol L^{-1} CTAB was chosen as the optimum.

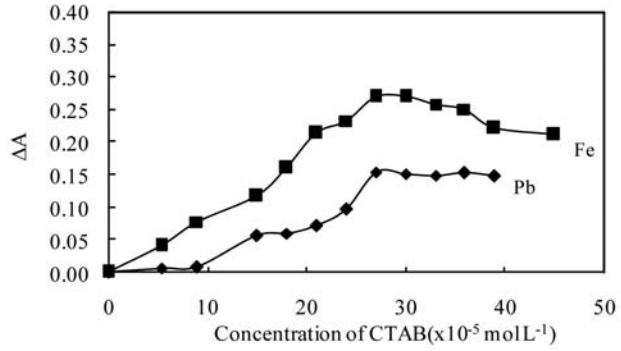

Fig. 5. Effect of concentration of CTAB on the extraction of Fe^{3+} and Pb^{2+} ions

Addition of salt can cause cationic surfactant solutions to separate into immiscible surfactant-rich and surfactant-poor phases. Several inorganic salts including NaCl, NaF, KNO_3, KBr and KI, were tested and KI was found as the best. Therefore, iodide was added to induce micelle growth and extraction of complex.

The effect of iodide concentration was studied in the range 0.003–0.03 mol L^{-1}, addition of 0.02 mol L^{-1} iodide sufficed for maximum extraction of the complexes and the signal and remained constant at higher concentrations. A concentration of 0.02 mol L^{-1} iodide was selected for further works (Fig. 6).

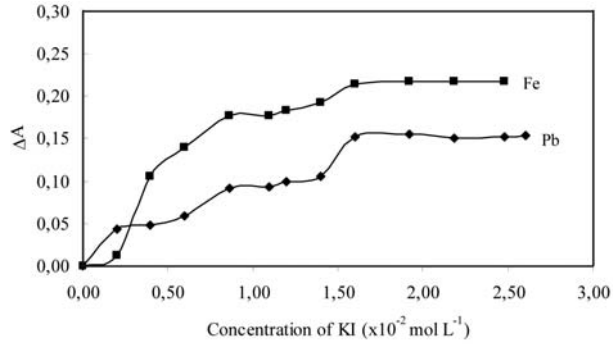

Fig. 6. Effect of concentration of KI on the extraction of Fe^{3+} and Pb^{2+} ions

Effect of time on the reaction and also on the CPE procedure was investigated. The results showed that complexation reactions were completed in 10 min. Also a 15 min centrifugation at 3800 rpm was found to be enough for successful CPE.

Because the surfactant-rich phase was precipitate, different solvents were tried so as to select the one producing the optimal results regarding sensitivity. Among methanol, ethanol, DMF, acetone and acetonitrile, DMF gave the best results due to high sensitivity and low overlapping of spectra of components. Therefore, DMF was chosen in order to have appropriate amount of sample for transferring and measurement of the absorbance of the sample and also a suitable preconcentration factor.

3. 2. Analytical Characteristics

Table 1 summarizes the analytical characteristics of the optimized methods, including regression equation, linear rang and limit of detection, preconcentration and improvement factors. The limit of detection, defined as C_L =

$3S_B/m$ (where C_L, S_B, and m are the limit of detection, standard deviation of the blank and slope of the calibration graph, respectively), were 0.020 ng $mL^{-1}$and 0.040 ng mL^{-1} for Fe^{3+} and Pb^{2+} ions, respectively. Because the amounts of Fe^{3+} and Pb^{2+} ions in 10 mL of sample solutions is measured after preconcentration by CPE in a final volume of 0.5 mL DMF, the solutions is concentrated by a factor of 20. The improvement factor, defined as the ratio of the slope of the calibration graph for the CPE method to that of the calibration graph in micellar media without preconcentration, were 22.0 and 47.1 for Fe^{3+} and Pb^{2+} ions, respectively.

3. 3. Selectivity

In order to study the selectivity of the proposed methods, the effect of various cations and anions was tested on the preconcentration and determination of 30 ng mL^{-1} Fe^{3+} and 30 ng mL^{-1} Pb^{2+} by the proposed methods under the optimum conditions. The results are summarized in Table 2. The tolerance limit was defined as the concentration of added ion that caused less than ± 3% relative error. The tolerable levels of the some metal ions are suitable for the separation and preconcentration of ions in the real samples examined present study.

3. 4. Applications

To evaluate the validity of the proposed methods for real sample analysis, the proposed procedures were applied to various real samples. The method was used for the determination of Fe^{3+} and Pb^{2+} ions in water samples (river water, tap water), vegetables including lettuce, spinach, cabbage, parsley, dill and tea, rice, chicken and fish samples. The results are given in Tables 3–5. The recovery of spiked samples is satisfactorily reasonable and was confirmed using addition method, which indicates the capability of the system in the determination of ions. A good agreement was obtained between the added and measured analyte amounts. The recovery values calculated for the added standards were always in the range 95–104 %, thus confirming the accuracy of the procedure and its independence from the matrix effects.

Table 1. Analytical features of the proposed method for determination of Fe^{3+} and Pb^{2+}

	Fe^{3+}	Pb^{2+}
Regression equation (n = 20)	ΔA = 0.011C+ 0.0093 r = 0.9993	ΔA = 0.0031C+ 0.0489 r = 0.9986
Regression equation before preconcentration(n = 11)	ΔA = 0.0005C–0.0223 r = 0.9981	ΔA = 0.0658C–0.0113 r = 0.9991
Linear range (ng mL^{-1})	0.05–43.00, (50.0–550.0)[a]	0.1– 40.0, (0.75–6.00)[a]
Limit of detection ($3SD_B$/m blank, ng mL^{-1}) (n = 5)	0.020	0.04
Preconcentration factor	20.0	20.0
Improvement factor	22	47.1

[a] Linear range before preconcentration (ng mL^{-1})

The relative standard deviation (R.S.D.) and relative error (R.E.) for five replicate measurements of 5.00 ng mL^{-1} of Fe^{3+} was 3.8% and 3.85% , for 30.00 ng mL^{-1} of Fe^{3+} was 2.60% and 1.60% and for 1.00 ng mL^{-1} of Pb^{2+} was 2.24% and 4.03% for 15.00 ng mL^{-1} of Pb^{2+} was 1.82% and 1.83%, respectively.

Table 2. Tolerance ratios of diverse ions on the determination of 30.0 ng mL^{-1} of Fe^{3+} and Pb^{2+} ions

Ion	Tolerance ratio (mol ratio)	
	Fe^{3+}	Pb^{2+}
K^+, Na^+, Ca^{2+}, Mg^{2+}, F^-, SO_4^{2-}, CN^-, Cl^-, NO_3^-, PO_4^{3-}	1000	1000
Ba^{2+}, Cd^{2+}, Zn^{2+}, Co^{2+}, Mn^{2+}	500	500
Ni^{2+}, Sr^{2+}, Cr^{3+}	150	150
Citrate and tartarate	80	80
S^{2-}	30	30
Ag^+	25	25
Hg^{2+}	10	10
Al^{3+}	2(50)[a]	2(50)[a]
Cu^{2+}	2(100)[b]	2(100)[b]
Fe^{3+}	–	1
Pb^{2+}	1	–

[a] After removal by NaF, [b] After removal by $Na_2S_2O_3$

Table 3. Determination of Fe^{3+} in the water samples by the proposed method

Sample	Fe^{3+} added (ng mL^{-1})	Fe^{3+} found[a] (ng mL^{-1})	Recovery (%)
Tap water	0	2.48 ± 0.02	–
	0.50	2.96 ± 0.02	96.0
	10.00	12.15 ± 0.02	96.7
	30.00	31.10 ± 0.03	95.4
River water	0	1.38± 0.02	–
	0.30	1.68 ± 0.02	100.0
	10.00	11.45 ± 0.03	100.7
	20.00	22.27 ± 0.02	104.4

[a] Mean ± S. D. (n = 3)

Table 4. Determination of Fe^{3+} in human hair, tea, spinach and liver samples by the proposed method

Sample	Fe^{3+} added (ng mL^{-1})	Fe^{3+} Found* (ng mL^{-1})	Recovery (%)	Fe^{3+} found** (μg g^{-1})
Human hair 1	–	1.28 ± 0.02		32.00
	0.30	1.59± 0.03	103.3	–
	6.00	7.36 ± 0.02	101.3	–
	20.00	22.05 ± 0.03	103.8	–
Human hair 2	–	5.66 ± 0.02	–	56.60
	0.30	5.95 ± 0.02	96.6	–
	6.00	11.50 ± 0.02	97.3	–
	20.00	26.35 ± 0.03	103.4	–
Human hair 3	–	4.18 ± 0.02	–	41.80
	0.50	4.69 ± 0.02	102.0	–
	10.00	13.77 ± 0.03	96.1	–
	30.00	35.36 ± 0.02	103.9	–
Black tea	–	12.30 ± 0.02	–	123.00
	1.00	13.38± 0.03	103.0	–
	15.00	26.65 ± 0.02	95.6	–
	30.00	42.07 ± 0.03	99.2	–
Green tea	–	11.43 ± 0.02	–	228.56
	1.00	12.40 ± 0.02	97.0	–
	15.00	25.77 ± 0.02	95.6	–
	30.00	41.12 ± 0.03	98.9	–
Spinach	–	15.43± 0.01		1.0
	1.00	16.44± 0.02	101.0	–
	10.00	24.93± 0.02	95.0	–
	20.00	35.42± 0.03	99.9	–
Liver	–	4.17 ± 0.02	–	104.25
	2.00	6.21 ± 0.02	102.0	–
	10.00	14.31 ± 0.03	101.4	–

* Fe contents in the final sample solutions; ** (μg g^{-1} in the dry sample)

Table 5. Determination of Pb^{2+} in foodstuff and chicken samples by the proposed method

Sample	Pb^{2+} added (ng mL^{-1})	Pb^{2+} Found* (ng mL^{-1})	Recovery (%)	Pb^{2+} found** (μg g^{-1})
Lettuce	–	17.18 ± 0.02	–	0.74
	1.00	18.20± 0.01	102.0	–
	10.00	27.49 ± 0.02	103.1	–
	20.00	37.57 ± 0.03	101.9	–
Dill	–	7.05 ± 0.02	–	0.36
	1.00	8.07 ± 0.01	102.0	–
	10.00	17.33 ± 0.02	102.8	–
	20.00	26.62 ± 0.02	97.8	–
Parsley	–	2.64 ± 0.02	–	0.32
	1.00	3.63 ± 0.02	99.0	–
	10.00	12.44 ± 0.03	98.0	–
	30.00	33.10 ± 0.03	101.5	–
Cabbage	–	4.09 ± 0.02	–	0.29
	1.00	5.07 ± 0.02	98.0	–
	10.00	14.02 ± 0.03	99.3	–
	30.00	33.82 ± 0.03	99.1	–
Rice	–	12.73 ± 0.02	–	1.02
	1.00	13.70 ± 0.01	97.0	–
	10.00	23.09 ± 0.02	103.6	–
	20.00	32.49 ± 0.03	98.8	–
Chicken	–	5.25 ± 0.02	–	0.17
	1.00	6.27 ± 0.01	102.0	–
	10.00	15.31 ± 0.02	100.1	–
	30.00	35.45 ± 0.03	100.6	–

* Pb^{2+} contents in the final sample solutions; ** (μg g^{-1} in the dry sample)

4. Conclusion

The proposed procedure gives a simple, high sensitive and low-cost spectrophotometric procedure for determination of Fe^{3+} and Pb^{2+} ions that can be applied to real samples. The surfactant has been used for preconcentration of Fe^{3+} and Pb^{2+} in water, and thus toxic solvent extraction, has been avoided. A comparison between the proposed method with the previously reported methods for preconcentration and determination of Fe^{3+} and Pb^{2+}[16,43–45] indicates that this method has a lower detection limit, wider linear range and is a convenient, safe, simple, rapid and inexpensive method for the determination of trace quantities of Fe^{3+} and Pb^{2+} ions to real samples.

5. References

1. M. Kuramochi, K. Tomioka, M. Fujinami, K. Oguma, *Talanta* **2006,** *68***,** 287–291.
2. R. Liu, P. Liang, *J. Hazard. Mater.,* **2005,** *152*, 166–171.
3. J. Chen, S. Xiao, X. Wu, K. Fang, W. Liu, *Talanta*, **2005**, *67***,** 992–996.
4. J. L. Manzoori; M. Amjadi; J. Abulhassani; *Anal. Chim. Acta*, **2009***, 644***,** 48–52.
5. S. Jaenicke, R. M. Sabarathinam, B. Fleet, H. Gunasingham, *Talanta* **1998** *45*, 703–711.
6. A. L. D. Comitre, B. F.Reis, *Talanta* **2005***, 65*, 846–852.
7. G. Fang, S. Meng, G. Zhang, J. Pan, *Talanta* **2001,** *54*, 585–592.
8. T. Tomiyasu, N. Yonehara, N. Teshima, T. Kawashima, *Anal. Chim. Acta* **1999** *394*, 55–63.
9. J. Q. Chen, W. Gao, J. F. Song, *Sens. Actuators, B* **2006***, 113*, 194–200
10. N. Teshima, S. Gotoh, K. Ida, T. Sakai, *Anal. Chim. Acta,* **2006***, 557,* 387–392.
11. S. Ohno, N. Teshima, T. Sakai, K. Grudpan, M. Polasek, *Talanta* **2006***, 68* 527–534.
12. E. P. Achterberg, T. W. Holland, A. R. Bowie, R. F. C. Mantoura, P. J., Worsfold *Anal. Chim. Acta,* **2001,** *442*, 1–14.
13. R. Karimi-Shervedani, A. Hatefi-Mehrjardi, A Asadi-Farsani., *Anal. Chim. Acta***, 2007***, 601*, 164–171.

14. R. N. M. J. Páscoa, I. V. Tóth, l A. O. S. S. Range, *Microchem. J.* **2009***, 93* 153–158.
15. F. M. V. Pereira, D. P. Pereira-Filho, M. I. M. S. Bueno, *J. Agric. Food Chem.* **2006,** *54*, 5723–5730.
16. U. Divrikli, A. Akdogan, M. Soylak, L. Elçi, *J. Hazard. Mater.* **2007***, 149*, 331–337.
17. F.A. Aydin, MM. Soylak, *Talanta*, **2007**, *73*, 134–141.
18. M. T. Naseri, M. R. Milani-Hosseini, Y. Assadi, A. Kiani, *Talanta,* **2008***, 75,* 56–62.
19. J. Li, F. Lu, T. Umemura, K. I Tsunoda, *Anal. Chim. Acta* **2000,** *419,* 65–72.
20. J. Li, S. Guo, Y. Zhai, E. Wang, *Anal. Chim. Acta* **2009***, 649*, 196–201.
21. M. Kass, A. Ivaska, *Talanta* **2002***, 58*, 1131–1137.
22. T. Pojanagaroon, S. Watanesk, V. Rattanaphani, S. Liawrungrath, *Talanta* **2002***, 58*, 1293–1300.
23. D. M. C. Gomes, M.A. Segundo, J.L.F.C. Lima, A.O.S.S. Rangel, *Talanta* **2005,** *66,* 703–711.
24. E. Y. Hashem, M.S. Abu-Bakr, S.M. Hussain, *Spectrochim. Acta A* **2003***, 59*, 761–769.
25. P. K. Tarafder, R. Thakur, *Microchem. J.* **2005***, 80,* 39–43.
26. M. S. D. Nezio, M. E. Palomeque, B. S. F. Band, *Talanta* **2004***, 63*, 405–409.
27. S. Ninan, A. Varadarajan, S.B. Jadhav, A.J. Kulkarni, S.P. Malve, *Spectrochim. Acta A* **1999,** *55*, 825–831.
28. Li J. Liang, C. B. Hung, *J. Colloid Interface Sci.* **2003**, *263*, 625–632.
29. H. Hoffmann, in: C. A. Herb, R. Prudhomme (Eds.), Structure and Flow in Surfactant Solutions, ACS Symposium Series, 578, American Chemical Society, Washington, DC, 1994, p. 2.
30. A. E. Vassiliades, in: E. Jungerman (Ed.), Cationic Surfactants, Marcel Dekker, New York, 1970, p. 387.
31. A Afkhami., T. Madrakian, H. Siampour, *J. Hazard. Mater.* **2006***, 138,* 269–272.
32. A. Afkhami, T. Madrakian, H. Siampour, *J. Braz. Chem. Soc.* 2006, *17*, 797–802.
33. A. Afkhami, T. Madrakian, E. Bozorgzadeh, M. Bahram, *Talanta 71* **2007,** 1103–1109.
34. T. Madrakian, A. Afkhami, A. Mousavi, *Talanta* **2007,** *71,* 610–614.
35. T. Madrakian, F. Ghazizadeh, J. *Hazard. Mater.* **2008***, 153,* 695–700.
36. M. Ghaedi, M.R. Fathi, A. Shokrollahi, S. Gharaghani, F. Ahmadi, M. Soylak, *Quimica Nova*, **2008**, *31*, 70–74.
37. J. A. Dean, Analytical Chemistry Handbook, McGraw-Hill Inc., **1995**, pp.14–33.
38. S. B. Deeming, C. W. Weber, *Am. J. Clin. Nutr.,* **1978**, 1175–1180.
39. K. S. Rao, T. Balaji, T. P. Rao, Y. Babu, G. R. K. Naidu, *Spectrochim. Acta B* **2002***, 57,* 1333–1138.
40. M. Ghaedi, A. Shokrollahi, A. H. Kianfar, A. S. Mirsadeghi, *J. Hazard. Mater.* **2008***, 154*, 128–134.
41. K. L. Cheng, K. Ueno, T. Imamura, CRC Hand book of organic analytical reagents, CRC Press, **1990**, pp. 45–50.
42. H. Xe-Wen, D. P. Poe, *Talanta* **1981,** *28*, 419–424.
43. S. Kagaya, Y. Araki, K. Hasegava, *Fresenius. J. Anal. Chem.* **2000***, 366*, 842–845.
44. L. Silva, P. Roldan, *J. Hazard. Mater.* **2009***, 161,* 142–147.
45. P. Liang, H. Sang, Z. Sun; *J. Colloid. Interface Sci.* **2006,** *304,* 486–490.

Povzetek

Proučevana je bila ekstrakcija z uporabo micel na osnovi kationskega surfaktanta CTAB iz vodnih raztopin. Metoda je osnovana na barvni reakciji Fe^{3+} in Pb^{2+} z brompirogalolom rdeče pri pH 5,0 in ekstrakcijo kompleksa s kationskim surfaktantom. Na osnovi izbranih optimalnih pogojev ekstrakcije in reakcije so bile določene analizne karakteristike predstavljene metode (meja zaznave, linearno območje, koncentrakcijski faktorji). Preizkušeni so bili tudi vplivi nekaterih motečih anionov in kationov. Metoda je bila uporabljena za določanje Fe^{3+} in Pb^{2+} v raznih vrstah zelenjave, čaju, rižu, človeških laseh, jetrih, piščančjem mesu in vodnih vzorcih.