



Application of rapid cloud point extraction method for trace cobalt analysis coupled with spectrophotometric determination



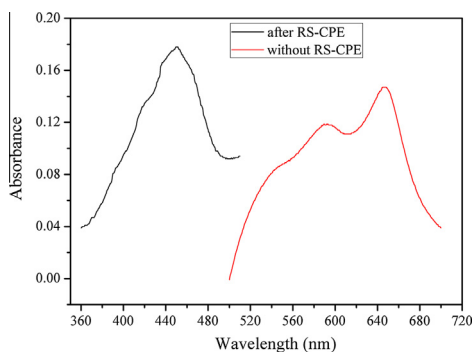
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HIGHLIGHTS

- The first application of RS-CPE coupled with UV–vis for Co detection.
- Conventional UV–vis accomplishes ultra-trace detection via the coupling.
- RS-CPE is novel and rapid method with impressive detectability and novelty.
- Be useful in less developed area without more advanced analytical instruments.

GRAPHICAL ABSTRACT



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ABSTRACT

In this work, the analytical performance of conventional spectrophotometer was improved through the coupling of effective preconcentration method with spectrophotometric determination. Rapidly synergistic cloud point extraction (RS-CPE) was used to pre-concentrate ultra trace cobalt and firstly coupled with spectrophotometric determination. The developed coupling was simple, rapid and efficient. The factors influencing RS-CPE and spectrophotometer were optimized. Under the optimal conditions, the limit of detection (LOD) was $0.6 \mu\text{g L}^{-1}$, with sensitivity enhancement factor of 23. The relative standard deviation (RSD) for seven replicate measurements of $50 \mu\text{g L}^{-1}$ of cobalt was 4.3%. The recoveries for the spiked samples were in the acceptable range of 93.8–105%.

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Introduction

Spectrophotometric analysis is one of classical methods of molecular spectroscopy analysis. This kind of analytical methods and instruments have been widely applied in analytical chemistry for decades and offer many appealing characteristics, including simple instrumentation, rapid response times, and easy operation [1]. With the rapid development of analytical chemistry, higher demand of analytical performance such as sensitivity, precision and

micro-consumption of sample volume has been put forward for analytical methods and apparatus. As one of classical instruments, the disadvantages of traditional spectrophotometer in analytical performance have been increasingly apparent. But compared with other advanced and expensive instruments, conventional spectrophotometer is very cost-effective and widely used even in some less developed area [2]. If solving the choke point of poor sensitivity, the instrument could be more useful. To couple some advanced preconcentration techniques with traditional spectrophotometer, trace or ultra-trace metal ions could be successfully detected [3,4]. This kind of research works have been relatively seldom carried out recently, such as single drop microextraction [5–7], solid

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phase extraction [8–10], cloud point extraction and surfactant-mediated extraction [11–14], dispersive liquid–liquid microextraction [15–17].

In this work, the coupling of rapidly synergistic cloud point extraction (RS-CPE) with conventional spectrophotometer was investigated to determine trace cobalt. RS-CPE has been established very recently by our group and considerably simplified and accelerated traditional CPE procedure [18–20]. Based on the work of Chang et al. [21], octanol was used as cloud point revulsant and synergic reagent to lower the cloud point temperature of surfactant Triton X-114 and assisted the subsequent extraction process. Without any heating units, RS-CPE could be accomplished in only 1 min at room temperature. In order to expand its application, cobalt was firstly pre-concentrated by this method and subsequently detected by spectrophotometer. Cobalt is known to be essential at trace levels to man, animals and plants for metabolic processes [22]. The determination of trace amounts of cobalt in natural waters is of great interest because it is important for living species as complex Vitamin B12. Toxicological effects of large amounts of cobalt include vasodilation, flushing and cardiomyopathy in human and animals [23]. In the previous works, many instrumental methods have been reported for the determination of cobalt, such as inductively coupled plasma–optical emission spectrometry (ICP-OES) [24], atomic fluorescence spectrometry (AFS) [22,25], capillary electrophoresis (CE) [26], electrothermal atomic absorption spectrometry (ET-AAS) [27,28], flame atomic absorption spectrometry (FAAS) [29] and spectrophotometry [30,31].

To the best of our knowledge, this was the first application of RS-CPE in UV–vis for the determination of trace cobalt in water samples. The developed coupling was very simple and rapid for extraction and determination. The analytical performance of conventional spectrophotometer was considerably improved and the application of RS-CPE was expanded. The main parameters influencing extraction and determination were investigated in detail. The characteristics and performance parameters of the method developed here were described below.

Experimental

Apparatus

UV–vis spectrophotometer Model TU-1901 (Beijing Puxi General Instrument Co., Ltd, Beijing, China) was used for the investigation and determination.

Phase separation was achieved via a centrifuge Model TDL-5-A (Shanghai Anting Scientific Instrument Factory, Shanghai, China) in 40 mL calibrated conical tubes. The pH values were measured by a pH-meter Model pHS-25 (Shanghai Hongyi Instrument Co., Ltd, Shanghai, China).

A laboratory pure water system Model DZG-303A (Chengdu Tangshi Kangning Science and Technology Development Co., Ltd, Chengdu, China) was used to prepare ultra pure water.

Reagents

Cobalt working standard solution was prepared daily by stepwise dilution from standard stock solution (1000 mg L^{-1}) in ultra pure water. Non-ionic surfactant Triton X-114 (TX-114) and octanol (Sinopharm Chemical Reagent Co., Ltd, Shanghai, China) were used as extractant and cloud point revulsant/synergic reagent, respectively. 1-(2-pyridylazo)-2-naphthol (PAN, The Sixth Factory of Chemical Reagent, Tianjin, China) was used as the chelating reagent for cobalt. Other chemical reagents including sulfuric acid, methanol, ethanol, tetrachloromethane (CCl_4), chloroform (CHCl_3),

acetone and tetrahydrofuran (THF) were all of analytical grade or better, as well as the reagents mentioned above.

Ultra pure water was used throughout to decrease blank.

Rapidly synergistic cloud point extraction (RS-CPE) procedure

The extraction procedure was schematically illustrated in the previous report [18]. For each sample, 40 mL analytical solution containing cobalt and chelating agent PAN were mixed in the solution. After adjusting the pH value, the proper volume of TX-114 and octanol were added into the conical tube. After manual shaking, the mixture solution became turbid by the effect of octanol. The extraction was accomplished in 1 min effectively. The complex of Co-PAN was captured by the micelle of TX-114 and dispersed octanol. After centrifugation, the turbid solution became pellucid and the micelle rich phase with octanol capturing complex was supernatant in the solution. The lower water phase was blotted up carefully and then diluting solvent was adopted to properly dilute the surfactant-rich phase to 3 mL, considering the sampling volume of spectrophotometer. After that, the resultant sample was transported to spectrophotometer for determination.

Sample preparation

Tap water was collected in our laboratory after flowing for about 5 min, river and lake water were gotten from Xier River and Erhai Lake (Dali, China). Waste water samples were collected from local factories. All the real water samples above were filtered through a $0.25 \mu\text{m}$ micropore membrane prior to use.

Results and discussion

Investigation of the absorption spectra of complex

In this work, spectrophotometric determination was coupled with the RS-CPE preconcentration. The maximum wavelength of absorption for the resultant sample was the basis of the quantification analysis. In the preliminary experiment, the resultant samples were conveyed to the spectrophotometer to measure the absorption curves, according to the experimental procedure described above. During the experiment, the cobalt concentration of $2000 \mu\text{g L}^{-1}$ was used for Co-PAN wavelength scan without

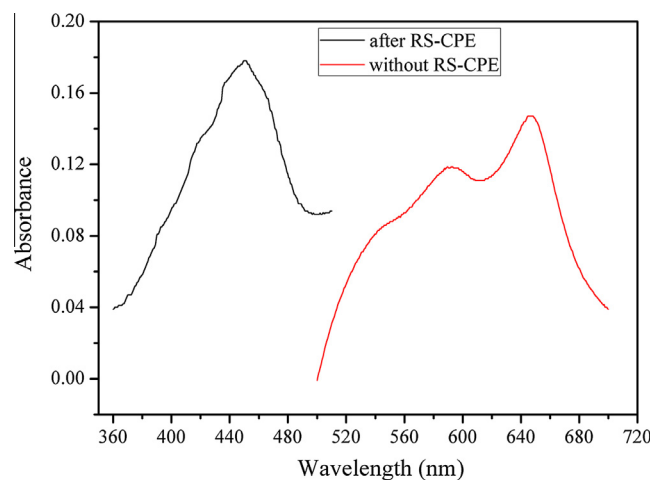


Fig. 1. Absorption spectra for the determination of Co with/without RS-CPE. RS-CPE conditions: Co, $200 \mu\text{g L}^{-1}$; PAN, $5.0 \times 10^{-5} \text{ mol L}^{-1}$; TX-114, 0.125% v/v; octanol volume, 1.0 mL; pH, 0.9; sample volume, 40 mL; dilution solvent, $1 \text{ mol L}^{-1} \text{ HNO}_3$ in methanol. Without RS-CPE conditions: Co, $2000 \mu\text{g L}^{-1}$; PAN, $5.0 \times 10^{-5} \text{ mol L}^{-1}$; pH, 0.9.

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