Periodate and iodate are important oxidants which can oxidize many inorganic and organic compounds. These ions have been extensively used for oxidation of metal ions, polyhydroxylated compounds and catalytic application at trace level [1, 2]. Periodate and iodate due to their special functions are essential micronutrients, markers of geochemically and biologically active processes, hazardous contaminants (in the form of radionuclides), and so on [3], they play an important biological and environmental role. The major route of environmental exposure to these oxihalides is drinking water [4]. Iodide ions may be found in brackish water and, to a lesser extent, in freshwater, and may form iodate during ozonization [5]. Therefore, the determination of these ions is required for a better understanding of biological and chemical processes for human health and environmental protection [5–7].

Spectrophotometric methods offer many appealing characteristics, including simple instrumentation, rapid response time and easy operation. These properties are highly desirable to the future design and development of portable analytical devices capable of quickly responding to trace levels of hazardous compounds in the field.

Different spectrophotometric methods for the determination of periodate and iodate in mixtures have been described in the literature. Sequential spectrophotometric methods have been used to determine of periodate and iodate using flow injection systems [8–10], differential kinetic methods [11, 12] and design of experimental conditions [13–15]. Simultaneous spectrophotometric methods have been used to determine periodate and iodate using chemometric programs, such as classical least square (CLS), principal component regression (PCR), partial least square (PLS), orthogonal signal correction (OSC-PLS), back-propagation artificial neural network (BP-ANN), radial basis function–artificial neural network (RBF-ANN) and principle component–radial basis function–artificial neural network (PC-RBF-ANN) [16, 17].

In recent decades, the development of preconcentration steps in order to be implemented prior to analytical determinations of trace level compounds has been explored in considerable depth. Separations and preconcentrations based on cloud point extraction (CPE) are becoming an important and practical application of the use of surfactants in analytical chemistry [18]. The technique is based on the property of most non-ionic surfactants in aqueous solutions to form micelles and become turbid when heated to the cloud point temperature. Above the cloud point temperature the micellar solution separates in a surfactant-rich phase of a small volume and in a diluted aqueous phase, in which the surfactant concentration is close to the critical micellar concentration (cmc). Any anayte solubilized in the hydrophobic core of the micelles will separate and

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become concentrated in the small volume of the surfactant-rich phase. The phase separation mechanism can be attributed to ethylene oxide segments in the micelle that repel each other at low temperatures, when they are hydrated, and that attract each other as the temperature increases due to dehydration. This effect causes a decrease in the effective area occupied by the polar group on the micelle surface, increasing the size of the micelle, which can be considered to become infinite at the cloud point, resulting in the phase separation [19].

The purpose of this study is to propose a method for the spectrophotometric determination of periodate and iodate after preconcentration in a simple CPE process. The method is based on their reaction with iodide and produces triiodide at two different conditions and the subsequent micelle-mediated extraction of the triiodide formed. To the best of my knowledge, there has been no report on the preconcentration of periodate and iodate using the cloud point extraction methodology.

EXPERIMENTAL

Apparatus. A Hitachi model 3310 UV-Vis spectrophotometer with 1-cm quartz cells (1.0 mL) was used for recording absorbance spectra. All spectral measurements were performed using the blank solution as a reference. A centrifuge with 10 mL calibrated centrifuge tubes (Hettich, Germany) is used to accelerate the phase separation process.

Reagents. All chemical reagents used were of analytical reagent grade, and triply distilled water was used throughout the experiments. Standard solutions of periodate and iodate were prepared by dissolving an appropriate amount of sodium periodate or sodium iodate (all from Merck) in water. A 0.10 M iodide solution was prepared by dissolving 0.75 g sodium iodide (Merck) in water and diluting to the mark in a 50 mL volumetric flask. Triton X-114 stock solution (2% w/v) was prepared by dissolving 2 g of concentrated solution in distilled water. A 0.01 M hydrochloric acid solution was prepared by appropriate dilution of concentrated hydrochloric acid (Merck).

Determination of periodate and iodate in mixture.
Two runs are needed for each sample.

Procedure 1. An aliquot of the solution containing 200–1000 ng of periodate and 40–4000 ng of iodate and 1 mL of 0.10 M iodide were transferred into a 10-mL tube. The solution was diluted to ca. 9 mL with water and then 1.0 mL of 2.0% (w/v) Triton X-114 solution was added. The solution was taken up to the mark with triply distilled water. Separation of two phases was accelerated by centrifugation for 5 min at 3500 rpm. The mixture was cooled in an ice-salt bath to increase the viscosity of the surfactant-rich phase, and the aqueous phase was easily decanted by simply inverting the tube. The surfactant-rich phase of this procedure was dissolved and diluted to 1.0 mL with ethanol and transferred to a 1.0-mL quartz cell for absorbance measurement at 358 nm. The dependence of the absorbance on the concentration of periodate was found to conform to the following equation:

\[ A_1 = a_1 + b_1 c_{\text{periodate}}. \]  

Procedure 2. An aliquot of the solution containing 20–3000 ng of periodate and 40–4000 ng of iodate, 1 mL of 0.10 M iodide and 1 mL of 0.01 M HCl was transferred into a 10-mL tube. The solution was diluted to ca. 9 mL with water and allowed to stand for 5 min at room temperature. Then 1.0 mL of 2.0% (w/v) Triton X-114 solution was added. The solution was taken up to the mark with triply distilled water. Separation of two phases was accelerated by centrifugation for 5 min at 3500 rpm. The mixture was cooled in an ice-salt bath to increase the viscosity of the surfactant-rich phase, and the aqueous phase was easily decanted by simply inverting the tube. The surfactant-rich phase of this procedure was dissolved and diluted to 1.0 mL with ethanol and transferred to a 1.0-mL quartz cell for absorbance measurement at 358 nm. The dependence of the absorbance on the concentration of periodate and iodate was found to conform to the following equation:

\[ A_2 = a_2 + b_2 c_{\text{periodate}} + b_2^{'2} c_{\text{iodate}}. \]

RESULTS AND DISCUSSION

Periodate and iodate react with iodide in acidic media to liberate iodine

\[ 11\Gamma + IO_4^- + 8H^+ \rightleftharpoons 4I_3^- + 4H_2O, \]  

\[ 8\Gamma + IO_3^- + 6H^+ \rightleftharpoons 3I_3^- + 3H_2O. \]  

Triiodide ion product shows an absorption spectrum with maximum absorbance at 352 nm. It was observed that addition of the neutral surfactant Triton X-114 makes the solution turbid. Therefore, the triiodide product can be extracted by CPE method. The absorption spectrum of triiodide ion in surfactant-rich phase shows a maximum absorbance at 358 nm. After separation of surfactant-rich phase, the absorbance was measured in 358 nm against a reagent blank as the reference (Fig. 1). Also, it was observed that the reaction of iodate with iodide showed induction periods that were dependent on pH and reagent concentration. The induction period increased with decreasing acid concentration. However, the reaction of periodate initiated immediately after mixing with iodide in neutral and acidic media. Because of these differences, periodate and iodate could be determined by choosing suitable conditions. Two simultaneous equations were solved to give the periodate and iodate concentrations. Two runs were performed for each sample. All conditions were the same for two runs, except for pH, which was different for each run. The pH for the first run was nearly 6.0. At this pH only periodate reacted with iodide. Therefore, the absorbance was proportional to periodate concentration. The pH for the second run was nearly 3.0. At