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Original Article

Rapid determination of trace level copper in tea infusion samples by solid contact ion selective electrode

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ABSTRACT

A new solid contact copper selective electrode with a poly (vinyl chloride) (PVC) membrane consisting of *o*-xylylenebis(*N,N*-diisobutyldithiocarbamate) as ionophore has been prepared. The main novelties of constructed ion selective electrode concept are the enhanced robustness, cheapness, and fastness due to the use of solid contacts. The electrode exhibits a rapid (< 10 seconds) and near-Nernstian response to Cu²⁺ activity from 10⁻¹ to 10⁻⁶ mol/L at the pH range of 4.0–6.0. No serious interference from common ions was found. The electrode characterizes by high potential stability, reproducibility, and full repeatability. The electrode was used as an indicator electrode in potentiometric titration of Cu(II) ions with EDTA and for the direct assay of tea infusion samples by means of the calibration graph technique. The results compared favorably with those obtained by the atomic absorption spectroscopy (AAS).

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1. Introduction

Tea prepared from dried leaves of the *Camellia sinensis* plant is the most widely consumed nonalcoholic beverage worldwide because of its aroma, taste, smell, and multiple positive health-promoting effects [1,2]. Tea is consumed in the form of infusions worldwide from ancient times as a delectable item or as a natural medicine. Tea infusions are usually prepared by soaking the cured leaves, leaf buds, and internodes into boiling water. The hot water extracts various classes of

organic compounds, certain amounts of major, minor, and trace elements including metals. The chemical components of tea leaves and their infusions have attracted an ever-increasing attention because of their relation to health and disease. For these reasons, determination of heavy metals such as arsenic, chromium, cadmium, lead, copper, etc. in tea and tea infusion samples are important [3–5].

Copper is an essential trace element in biological systems and in living organisms where it serves as a cofactor in at least 30 important enzymes [6]. Despite copper being an essential

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element for humans, it is also toxic at elevated concentrations and is known to cause gastrointestinal catarrh, Wilson disease, hypoglycemia, and dyslexia [7,8].

There are several techniques, which have been used for the determination of copper in solution including flame atomic absorption spectroscopy (FAAS) [9], stripping voltammetry [10,11], inductively coupled plasma-mass spectrometry (ICP-MS) [12], spectrophotometry [13], and chromatography [14]. Most of the mentioned methods have drawbacks such as high cost of equipment and expensive materials, time-consuming, and complicated operation.

Application of ion-selective electrodes (ISEs) continues to be an interesting area of analytical research [15–17]. A number of Cu(II) selective electrodes have been developed using solid membranes of insoluble salts of copper, copper chelates, macro cyclic polyethers, noncyclic neutral ionophores containing dithiocarbamate groups, Schiff bases, calixarenes, sol-gel-coated wire, and some liquid membrane sensors using Cu(II) complexes have also been reported [18–28]. Among these ISEs, the solid-contact ones (SC–ISEs) have attracted extensive attention for the past few years [29,30]. Solid contact electrodes offer several advantages over the conventional electrodes regarding their simple construction, robustness, miniaturization, lower cost of production, and no risk of inner filling solution leakage problem. Furthermore, this type of electrode allows for low detection limit, wide measuring range, and working pH range [31–35].

In this paper, a new solid contact copper selective electrode with a PVC membrane consisting of *o*-xylylenebis(*N,N*-diisobutyldithiocarbamate) as ionophore has been prepared as an alternative copper selective electrode. The potentiometric properties of the electrode was investigated and applied to the assay of tea infusion samples. However, it has been demonstrated that the solid contact improved the analytical parameters of the electrode such as, response time and working pH range.

2. Methods

2.1. Materials

Tetrahydrofuran (THF), high molecular weight poly(vinyl chloride) (PVC), 2-nitrophenyl octyl ether (NPOE), potassium tetrakis (*p*-chlorophenyl) borate (KTpClPB), *o*-xylylenebis(*N,N*-diisobutyldithiocarbamate), and graphite were purchased from Fluka (Buchs, Switzerland). Epoxy (Macroplast Su 2227) and hardener (Desmodur RFE) were purchased from Henkel (Istanbul, Turkey) and Bayer AG (Darmstadt, Germany). All solutions were prepared from analytical-reagent grade salts using distilled deionized water. The chloride or nitrate salts of all cations (from Merck, Darmstadt, Germany) were used in all instances.

2.2. Apparatus and electrochemical measurements

A laboratory-made computer-controlled high-input impedance eight-channel potentiometric system was used for the potential measurement. The data output was recorded by a home-made software program. All the electromotive forces (Emf) were

measured relative to a saturated Ag–AgCl reference electrode (Gamry, Warminster, PA, USA) in solutions stirred with a magnetic stirrer. The pH of the solutions was adjusted by using a glass pH electrode (SI Analytics GmbH, Mainz, Germany) with a Jenway 3040 model Ion Analyzer (Bibby Scientific Limited, Staffordshire, UK). Solutions at required concentrations were homogenized using an Ultrasonic LC30 stirrer (Elma Hans Schmidbauer GmbH & Co. KG Kolpingstr, Singen, Germany).

Potentiometric measurements were carried out with the following cell assembly: SC–ISE | test solution | Ag–AgCl, KCl (saturated). The cell consists of SC–ISE as the indicator electrode, a saturated Ag–AgCl reference electrode, and a magnetic stirrer. The performance of the electrode was investigated by measuring the Emf's of copper nitrate solutions prepared in the concentration range 10^{-1} – 10^{-7} mol/L by serial dilution. All measurements were carried out at room temperature. Reference and indicator electrodes were washed with deionized water and dried with adsorbent tissue before each measurement of the solutions.

A Unicam 929 Model (Analytical Technology Inc., Collegeville, PA, USA) flame atomic absorption spectrophotometer, equipped with an air–acetylene flame burner and a deuterium continuous source background corrector was used for the determination of Cu(II). All operating conditions were as follows: wavelength, 324.8; lamp current (mA) 5, and slit width 0.5 nm.

Elga PureLab Option Q Water Purification System (Elga Process Water, Buckinghamshire, UK) was used for the purification of the water.

2.3. Electrode construction

A solid contact copper selective electrode was constructed as described in our previous works [35,36]. A conductive material was prepared by mixing 50% (w/w) graphite, 35% (w/w) epoxy, and 15% (w/w) hardener in sufficient THF. The mixture was allowed to stand in air until the appropriate viscosity was attained. A shielded copper wire was dipped into this mixture a few times to obtain a uniform solid contact with coating thickness of ~0.2 mm and then allowed to stand overnight in room temperature.

The ion selective membrane contained ionophore: PVC:KTpClPB:NPOE in the ratio of 6.9:57.2:1.6:34.9 (w/w; mg). The membrane solution was prepared by dissolving of the membrane components in 2.0 mL THF. Solid contacts were dipped into the membrane solution at least three times and then coated membranes were allowed to dry in air for at least 1 day. The schematic procedure for the preparation of solid contact electrode is shown in Figure 1. The dried membrane electrodes were soaked in a 1×10^{-2} mol/L $\text{Cu}(\text{NO}_3)_2$ solution for 1 day before use. The prepared electrodes were stored in laboratory conditions, when not in use. Before individual measurement process, the electrodes were reconditioned for at least half an hour in a 1.0×10^{-2} mol/L $\text{Cu}(\text{NO}_3)_2$ solution. Three identical electrodes were prepared and subjected to the same studies.

2.4. Sample preparation and determination

Four different tea samples were bought in a market place. For the preparation of tea infusion sample; extraction was

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