Determination of Lead in Cosmetic Sampels Using Coated Wire Lead (II) Ion Selective Electrode Based On Phyropillite

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Abstract-A coated wire ion selective electrode (CW-ISE) based on pyrophyllite for detection lead (II) in cosmetic samples was developed. CW-ISE has easy measurement for field analysis, good selectivity and sensitivity. The CW-ISE's membrane consist of mixture an inorganic material of pyrophyllite as ionophore, polyvinylchloride (PVC) as the matrix polymer, activated carbon and dioktilftalat (DOP) as a plasticizer dissolved in tetrahydrofuran (THF) solvent (1:3 w/v). It showed a good Nernstian slope of 29.33 mV/decade concentration, a lead linear range concentration between 10-1- 10-5 M, and detection limit of 8.054 x10-6 M (equal with 1.669 ppm of lead). And could be used optimally at pH 5 and showed a good selectivity for interfering ions (sodium ion, mercury ion and chromium ion) with selectivity order of lead >mercury>chromium>sodium. The CW-ISE Lead (II) based on pyrophyllite could be applied for determination lead in cosmetic samples, with precision of 95.42% and accuracy of 96.49%. Based on the statistical t test with 5% confidence limit, the potentiometric method and Atomic Absorption Spectrophotometry method had no significant differences.

Keywords- lead, pH, selectivity, potentiometric method,cosmetic sampels

INTRODUCTION

Lead as heavy metals are accumulates not only in the air, water, soil and even in food products and cosmetics found a real lead. In water, the threshold of lead exposure of 0.01 mg / L, in the air lead levels were still within the threshold for the ambient air of <1μm , whereas in food products and cosmetics is not allowed contain lead. Nevertheless there are still many food products and cosmetics samples that contain lead. Cosmetics is a substance or preparation used in the human body and the outside serves to beautify, embellish, perfume, or to change the appearance of [1]. One type of make up is a cosmetic preparation lipstick. This cosmetic contains a wide range of dyes pigments used on the lips. Attractive dye usually contain metals that are harmful to our bodies and can damage the digestive system. Requirements of safe cosmetics lipstick is used primarily metal-metal content should be negative. Metals found in samples of lipstick, among others, cadmium (Cd), chromium (Cr), arsenic (Ar), mercury (Hg), rhodamine-B, and lead (Pb)[2]. Lead accumulates in the body is quite dangerous than not digested in the excretory system consequently can accumulate in the body and may cause central nervous system, kidney function, inhibiting the formation of hemoglobin, and can affect the level of intelligence (IQ) in children[3][4][5].

Coated wire ion selective electrode (CW-ISE) is a kind of potentiometric sensor which has become increasingly important in the electrochemical analysis system. The analysis technology of CW-ISE is based on the relationship between electrode potential and ion activity so as to performa detection of ions. There are several advantages such as high selectivity, high sensitivity,quick analysis, low cost, wide variety of tested ions, and little sample size in this technology[6][7]. Ion selective membrane is the key component of an ion-selective electrode. Polyvinylchloride (PVC) is generally chosen as the preferred polymeric base for ion-selective membranes[8][9]. However, the utility of ion selective electrodes (CW-ISEs) is being increasingly realized by analytical chemists in view of the rapid growth of industry and technology all over the world, as they represent a rapid, accurate and low-cost method of analysis[10][11][12][13][14].

Therefore, in this study developed a coated wire ion selective electrode (CW-ISE) based on pyrophyllite for detection lead (II) in cosmetic samples. CW-ISE has easy measurement for field analysis, good selectivity and sensitivity. The CW-ISE lead (II) with an active material modification (ionophores) membranes using inorganic material is pyrophyllite. Pyrophyllite is a kind of natural material whose existence is abundant so that easy to obtain. Based on the research of Pribadi[15] , 2008 pyrophyllite has the ability as a cation exchanger as mineral type clays such as pyrophyllite have active sites on the surface[16]. Active sites on pyrophyllite is most reactive to low pH , and therefore has the potential to be used as active ingredients (ionophore) membrane in the manufacture of CW-ISE lead (II).

Membrane support material used in the manufacture of CW-ISE lead (II) based on pyrophyllite are PVC as the matrix polymer, plasticizer of dioktilftalat (DOP)and activated carbon in the solvent tetrahydrofuran (THF). On research previously has been made CW- ISE lead (II) based on Pyrophyllite that already characterized. The optimum composition of the CW-ISE lead (II) based on pyrophyllite obtained by comparison pyrophyllite 49%: 3% activated carbon: 16% PVC: DOP 32% (w/w) in THF solvent. The electrode showed a good *Nernstian* response of 29.33 mV/decade, a linear concentration range of lead $1x10^{-1}$ M to $1x10^{-5}$ M with detection limit in the order of $8.054\ x10^{-6}$ M (equal with 1.669 ppm of lead). The stable potentiometric signals are obtain within a short time periode of 1 min and could be used optimally at pH 5. The CW-ISE Lead (II) showed a good selectivity for interfering ions (sodium ion, mercury ion and chromium ion) with selectivity order of lead >mercury>chromium>sodium. The CW-ISE Lead (II) based on pyrophyllite could be applied for determination lead in cosmetic samples, with precision of 95.42% and accuracy of 96.49%. Based on the statistical t test with 5% confidence limit, the potentiometric method and Atomic Absorption Spectrophotometry method had no significant differences.

RESEARCH METHODS

1) Materials and Equipment

The materials used in this study are $Pb(NO_3)_2$ pa (EMerck), a solution of CH_3COOH pa (EMerck), CH_3COONa pa (EMerck), $Hg(NO_3)_2$ pa (EMerck), HNO_3 50% v/v p.a (EMerck), $Cr(NO_3)_3.9H_2O$ p.a (EMerck) and aquadest.

Equipment used in this study are : CW-ISE lead (II) based on pyrophyllite have in characterization, electrode Ag/AgCl type HI5313 Hanna, potentiometer (Schoot Geräte CG model 820), pH meters (Hanna), alligator clamps, analytical balance (Adventurer AR model 2130), centrifuges, whatman No.40 filter paper, oven, desiccator, the sample bottles, hot plate, and glass equipment

2) Experimental

a. Fabrication CW-ISE lead (II) based on pyrophyllite

Pyrophyllite (active ingredient membrane or ionophore membranes) were prepared before use. The preparation is chemically and physics activated. Chemical activated is doing with 2 M HCl was added immersion results filtered and rinsed with distilled water until free of Cl^- ions. Physical activation was doing by heating at high temperature with a temperature of 550 ° C for 4 hours. Membrane preparation was done by mixing the constituent materials pyrophyllite the membrane, activated carbon, PVC, DOP and solvent THF[17]. Membrane that is attached to the wire Pt mixed and then dried in the open air and then do the heating at a temperature of 50 ° C for 12 hours.

b. Determination of Lead (II) in Cosmetic samples using CW-ISELead (II) based on pyrophyllite

Measurements were performed by taking a sample of each 2 mL of sample already in preparation [4], and then added acetate buffer pH 5 and pH to 5, was transferred to 100 mL pumpkin drinks with distilled water, shaken until homogeneous. 25 mL samples were taken, then the resulting potential responses recorded and performed repetitions 3 times. The data obtained were compared with measurements using Atomic Absorption Spectrophotometry (AAS).

RESULTS AND DISCUSSION

a. Fabrication CW-ISE lead (II) based on pyrophyllite

As for the basic characterization CW-ISE Lead (II) based on pyrophyllite produces Nernst Factor of 29.33 mV / decade of concentration, with a linear concentration range of 10^{-5}-10^{-1} M and is able to detect cations lead up to 8.054 x 10^{-6} mol / L, equivalent to 1,669 ppm of lead. The CW-ISE Lead(II) can be used optimallay at pH 5 and have good selectivity for interfering ions sodium ion, kromium ion and mercury ion.

b. Determination of Lead (II) in Cosmetic samples using CW-ISELead (II) based on pyrophyllite

Determination of lead (II) in the cosmetic samples can be done with the potentiometric methods using Coated Wire Ion Selective Electrode (CW-ISE) Lead (II) based on pyrophyllite, is expected to be an alternative methods to replace Atomic Absorption Spectrophotometry (AAS) methods in analysis. In this study, the sample used is two kind of lipstick sample (sample A and sample B), where both samples were tested with both methods, to determine the accuracy and precision of lead (II) in a sample of both methods lipstick.

Results of measuring the concentration of lead (II) in the two samples showed that the potentiometric methods use the CW-ISE Lead (II) based on pyrophylite and methods of analysis used Atomic Absorption Spectrophotometry (AAS), can be seen in Table 1.

Table 1. Comparison of Potentiometric methods with AAS methods

	Potentiometric Methods Methods			AAS	
Sample precision	$[Pb^{2+}]$	% Accuracy	% Precision	$[Pb^{2+}]$	%
Sample A	3,22 ppm	94,747	92,11	3,40 ppm	94,120
Sample B	7,25 ppm	96,49	95,42	7,56ppm	98,995

From Table 1. Comparison of Potentiometric methods with AAS methods, it can be seen that different between precision and accuracy, where the AAS methods have precision higher than the potentiometric methods. Based on the statistical t test with 5% confidence limits, the results of the two methods was not significantly different. Its mean potentiometric methods using CW-ISE Lead (II)based on pyrophyllite can be used as an alternative methods to analysis lead(II) in cosmetics samples, in addition to methods of atomic absorption spectrophotometry.

CONCLUSION

The result showed that CW-ISE Lead (II) have a good Nernstian with Nernst factor values of 29.47 mV / decade of concentration. The effect of foreign ions to the performance of CW-ISE lead (II) was determined by measuring potential response of lead (II) with mixed solution methods have a good selectivity with not bothered by interfering ions (sodium, mercuriy and chromium). The CW-ISE Lead (II) based on pyrophyllite could be applied for determination lead in cosmetic samples, with precision of 95.42% and accuracy of 96.49%. Based on the statistical t test with 5% confidence limit, the potentiometric methods and Atomic Absorption Spectrophotometry methods had no significant differences.

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