DETERMINATION OF MERCURY(II) USING ZINC/ ALUMINIUM LAYERED DOUBLE HYDROXIDE-3(4-HYDROXYPHENYL)PROPIONATE MODIFIED MULTI-WALLED CARBON NANOTUBE PASTE ELECTRODE

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ABSTRACT

This study describes the preparation of chemically modified multi-walled carbon nanotube paste electrode with zinc/aluminium layered double hydroxide-3(4-hydroxyphenyl)propionate nanocomposite (Zn/Al-LDH-HPP) for determination of Hg(II) by cyclic voltammetry method. Experimental parameters influencing the voltammetric response including percentage of modifier, type of supporting electrolyte, pH and scan rate were examined and optimized. The results show that optimum conditions of the voltammetric response were obtained at 2.5% (w/w) of Zn/Al-LDH-HPP, 0.5 M potassium chloride, pH 5.0 and scan rate of 100 mV s^{-1}. A linear range was obtained within the concentration of 1.0×10^{-9} to 1.0×10^{-7} M Hg(II) and 1.0×10^{-7} to 1.0×10^{-3} M Hg(II) with the limit of detection 5.0×10^{-10} M Hg(II). The presence of 25-folds concentration of most metal ions did not interfere the anodic peak current except Zn(II). In conclusion, the proposed modified multi-walled carbon nanotube paste electrode showed a high sensitivity, good selectivity, repeatability and reproducibility, making it suitable for the determination of Hg(II) in marine animal samples, for instance, fish and shellfish.

PENENTUAN MERKURI(II) MENGGUNAKAN ELEKTROD PES NANOTIUB KARBON BERBILANG DINDING TERUBAH SUAI ZINK/ALUMINIUM LAPISAN BERGANDA HIDROKSIDA-3(4-HIDROKSIFENIL)PROPIONAT

ABSTRAK

Kajian ini menghuraikan penyediaan elektrod pes nanotiub karbon berbilang dinding terubah suai secara kimia dengan nanokomposit zink/aluminium lapisan berganda hidroksida-3(4-hidroksifenil)propionat (Zn/Al-LDH-HPP) bagi penentuan Hg(II) melalui kaedah voltammetri kitaran. Parameter kajian yang mempengaruhi gerak balas voltammetrik termasuk peratusan pengubah suai, jenis elektrolit penyokong, pH dan kadar imbasan telah dikaji dan dioptimumkan. Dapatan menunjukkan bahawa keadaan optimum gerak balas voltammetrik diperolehi pada 2.5% (w/w) Zn/Al-LDH-HPP, 0.5 M kalium klorida, pH 5.0 dan kadar imbasan 100 mV s^{-1}. Julat linear telah diperoleh di antara kepekatan 1.0 x 10^{-9} hingga 1.0 x 10^{-7} M Hg(II) dan 1.0 x 10^{-7} hingga 1.0 x 10^{-3} M Hg(II) dengan had pengesanan 5.0 x 10^{-10} M Hg(II). Kehadiran 25 kali ganda kepekatan kebanyakan ion logam tidak mengganggu arus puncak anodik kecuali Zn(II). Kesimpulannya, elektrod pes nanotiub karbon berbilang dinding terubah suai yang dicadangkan menunjukkan kepekaan yang tinggi, kepilihan yang baik, kebolehulangan dan kebolehhasilan, menjadikannya sesuai untuk penentuan Hg(II) dalam sampel hidupan laut, misalnya ikan dan kerang.

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LIST OF ABBREVIATIONS

ΔE_p	Difference in peak potential
AAS	Atomic absorption spectrometry
AdSV	Adsorptive stripping voltammetry
ASV	Anodic stripping voltammetry
CMCNTPE	Chemically modified carbon nanotube paste electrode
CMCPE	Chemically modified carbon paste electrode
CME	Chemically modified electrode
CNT	Carbon nanotube
CNTPE	Carbon nanotube paste electrode
CPE	Carbon paste electrode
CSV	Cathodic stripping voltammetry
CV	Cyclic voltammetry
CVAAS	Cold vapor atomic absorption spectrometry
DME	Dropping mercury electrode
DPASV	Differential pulse anodic stripping voltammetry
DPV	Differential pulse voltammetry
E^0	Formal reduction potential
E_{pa}	Anodic peak potential
E_{pc}	Cathodic peak potential
FTIR	Fourier transform infrared
GC	Glassy carbon
GCE	Glassy carbon electrode

HMDE	Hanging mercury drop electrode
I_{pa}	Anodic peak current
I_{pc}	Cathodic peak current
ISE	Ion-selective electrode
LDH	Layered double hydroxide
MWCNT	Multi-walled carbon nanotube
NPV	Normal pulse voltammetry
SEM	Scanning electron microscopy
SWCNT	Single-walled carbon nanotube
SWV	Square wave voltammetry
Zn/Al-LDH-HPP	Zinc/aluminium layered double hydroxide-3(4-hydroxyphenil)propionate

CHAPTER 1

INTRODUCTION

1.1 Elemental Mercury

Mercury is an element with the symbol Hg (from Greek: hydrargyrum) and known as quicksilver. Mercury is a metal with the atomic number 80 and atomic mass 200.59. It can be in the form of salts with two oxidation states Hg^{+} (mercurous) and Hg^{2+} (mercuric). Mercuric salts are more common than mercurous salts in the environment. Mercury with high purity is a dense, silvery white liquid with very low melting point of 234.321 K or -38.829 ^{o}C. Mercury occurs naturally in the earth's crust especially in the form of sulfides. The non-toxic red sulfide (cinnabar) is the common ore of the mercury and may contain up to 70% mercury.

Some inorganic mercury compounds are used in various fields. Mercury(II) chloride is used as a disinfectant in medicine and as a fungicide in agriculture. Calomel is used in medicine, in pyrotechnics and as a catalyst. Thiomersal is used as an antiseptic and antifungal agent in medicines and vaccines. Mercury is used as an antifouling agent in paints for ships, as a slimicide in paper production, as a component of pesticides and as bacterial agent in the making of amalgam dental fillings.

Mercury can be released into the environment by human activities such as mining, metal smelting, combustion of fossil fuels, waste disposal and industrial activities. Mercury can also be released into the environment by volcanoes and by natural evaporation from the ocean and land surfaces. Organic mercury compounds released into the environment are usually broken down to elemental mercury or mercuric compounds (Holmes, James, & Levy, 2009; Nordberg, Fowler, Nordberg, & Friberg, 2007; Virtanen, Rissanen, Voutilainen, & Tuomainen, 2007).

Gold mining is one of the largest sources which contribute mercury contamination in the air, water and ground with various forms of mercury. Mercury pollution from gold mining tends to increase due to the high price of gold. Besides, secondary mercury pollution from other sources, for instance, fossil fuels, dental amalgams, fluorescent lighting and incineration of medical and municipal waste are decreasing due to elimination of mercury and environmental regulations (Kozin & Hansen, 2013).

The most form of mercury spreading in the oceans is Hg^{2+} ion (inorganic mercury). This ion then reacts with organic substances and anaerobic microorganisms to become organic mercury such as methylmercury and dimethylmercury, wherein both of them are known as toxic substances. Methylmercury and dimethylmercury easily dissolve in water and they are absorbed by microorganisms. These microorganisms are consumed by small fish, which in turn become food for larger fish. Mercury is also present in the meat of fowl and grazing animals, like sheep and cows. Both fish and meat are consumed by human, therefore mercury is able to enter in the human body and accumulate via this food chain (Kozin & Hansen, 2013).

All forms of mercury are harmful to mammalian species, including human. The level of the adverse effects caused by mercury depends on several factors like the time, the duration and the route of exposure (Nordberg et al., 2007). Hazard effects of mercury have been presented in the cardiovascular system, motor system, renal system, immune system, reproductive system, gastrointestinal system, liver, kidneys, and nervous system (Nordberg et al., 2007; Zahir, Rizwi, Haq, & Khan, 2005).

There are two cycles involved in the environmental transport and distribution of mercury. Figure 1.1 shows the cycles of the mercury in the environment. The first cycle is global scope which involves the atmospheric circulation of elemental mercury vapor from sources on land to the oceans. The second cycle is local scope which involves methylation of inorganic mercury particularly from anthropogenic sources (Boening, 2000). Metallic mercury (Hg^0) from hydrosphere (sea, rivers, lakes and ground water) and geosphere (soil) evaporates to the atmosphere and then returns to the Earth's surface via the rainfall.

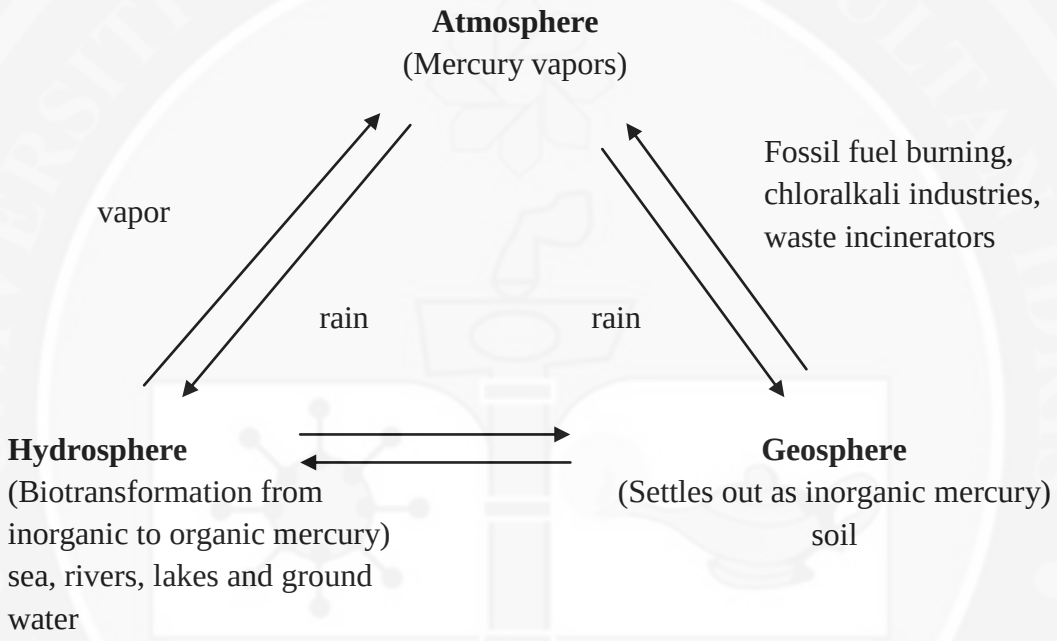

Figure 1.1. Cycles of the mercury in the environment.

1.2 Layered Double Hydroxide

Layered double hydroxide (LDH) is a group of synthetic two-dimensional nanostructured anionic clays (Li & Duan, 2006). It is called anionic clays because of its unusual properties of LDH exchange easily of interlayer anions. Hydrotalcite [$Mg_6Al_2(OH)_6CO_3 \cdot 4H_2O$] is one of the material group of LDH, which is naturally occurring in the environment, therefore LDH is known as hydrotalcite-like materials (Tonelli, Scavetta, & Giorgetti, 2013). The basic layer structure of a LDH is based on brucite [$Mg(OH)_2$], which consists of magnesium ions surrounded approximately octahedrally by six hydroxide ions, hence LDH is also known as brucite-like layer (Duan & Evans, 2006). Figure 1.2 shows the structure of a LDH.

The LDH can be fabricated by replacement of the divalent metal cations, coordinated octahedrally by hydroxyl groups in a brucite lattice, by trivalent metal cations, so that the layers obtain a positive charge then it is balanced by intercalation of organic or inorganic anions between the layers, and water molecule also usually occupy any free space in the interlayer area. The general formula of LDH is $[M^{2+}_{1-x}M^{3+}_{x}(OH)_2]^{x+}(A^{n-})_{x/n} \cdot mH_2O$, where M^{2+} is divalent cations (such as Mg^{2+}, Fe^{2+}, Co^{2+}, Cu^{2+}, Ni^{2+}, or Zn^{2+}) and M^{3+} is trivalent cations (such as Al^{3+}, Cr^{3+}, Ga^{3+}, Mn^{3+} or Fe^{3+}); A is an interlayer anion (such as CO_3^{2-}, SO_4^{2-}, Cl^- or NO_3^-) or an organic species; the value of x is equal to the molar ratio of $M^{2+}/(M^{2+} + M^{3+})$ and is usually in the range of 0.2–0.33; m is the number of water molecules located in the interlayer region together with anions and n^- is charge on interlayer ion (Duan & Evans, 2006).

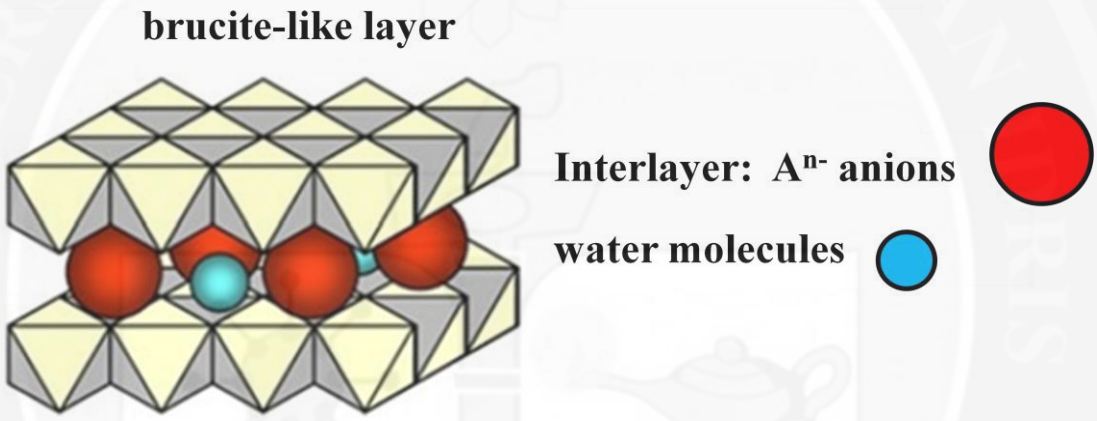

Figure 1.2. Structure of a LDH (Tonelli et al., 2013).

The LDH has a lot of physical and chemical properties which are astonishingly similar to those of clay minerals, such as the layer structure, the widely chemical compositions (due to modifiable isomorphous substitution of metallic cations), the modifiable layer charge density, the ion-exchange properties, the reactive interlayer area and the colloidal properties. These properties make LDH known as clay-like (Bergaya, Theng, & Lagaly, 2006).

The LDH has great interest for academic and industrial due to its several potential applications. One of the noteworthy properties of LDH is that the interlayer anions can be exchanged easily with numerous organic and inorganic charged compounds (Rives, 2001). LDH can be used for removal of harmful anions from liquid (Abdolmohammad-Zadeh, Rezvani, Sadeghi, & Zorufi, 2011; Goha, Lim, Banas, & Dong, 2010; Violante, Pucci, Cozzolino, Zhu, & Pigna, 2009) owing to the permanent positive charge, the high capacity of anion-exchange, the large surface area, the good thermal stability and the water-resistant structure (Tonelli et al., 2013). Mixed cation oxides, which are formed after thermal decomposition of LDH, can be used as catalysts (Basile et al., 2009) for a wide range of catalytic processes. The interlayer of LDH can act as a two-dimensional matrix for diverse chemical reactions (Rives, 2001). LDH also shows its potential as materials for several practical applications in pharmaceutics (Hoyo, 2007; Perioli et al., 2011; Trikeriotis & Ghanotakis, 2007), photochemistry (Musumeci et al., 2010; Shi, Wei, Evans, & Duan, 2010), polymer additives (Manzi-Nshuti et al., 2009) and electrochemistry (Yang et al., 2013; Yin, Cui, Ai, Fan, & Zhu, 2010). This is owing to its high versatility, easily modified properties and low cost, which make it feasible to create materials designed to execute definite requirements (Duan & Evans, 2006).

Recently, inorganic materials, like zeolites, clays and microporous, are widely applied in modified electrodes besides organic polymers. Inorganic materials have better stability, tolerance to high temperatures and oxidizing conditions, and chemical inertness, compare to organic polymers, therefore they are interesting as replacements of organic polymers (Duan & Evans, 2006). LDH clay-like is one of the inorganic materials that has been used as modifier in the electrode, known as chemically modified electrode (CME). Numerous studies have shown that LDH is a good material for construction of sensor based on CME (Guo, Zhang, Evans, & Duan, 2010; Mousty, 2010; Mousty, Vieille, & Cosnier, 2006). CME confines the analyte to a small amount close to the electrode surface during the preconcentration step, so that the low level of analyte can be measured. CME is greatly applied to electroanalysis because it has electrocatalytic properties that enhance the sensitivity and selectivity of the measurement (Tonelli et al., 2013).

Several types of electrodes modified with LDH show electrocatalytic activity. Biosphenol A was oxidized at glassy carbon electrode modified with Mg-Al-CO_3 LDH (Yin et al., 2010) and at glassy carbon electrode modified with Mg-Al LDH that functionalized with sodium dodecyl sulfonate (Yin et al., 2011). A nanomaterial Mg-Fe-Ni LDH, prepared by coprecipitation, is employed for immobilization of glucose oxidase on glassy carbon electrode. This CME shows good electrochemical performances with regard to the determination of glucose, like fast response, high sensitivity with low detection limit, wide range of linear concentration and long-time stability (Xu et al., 2011). A gold electrode modified with Zn-Al LDH by spreading a colloidal suspension to get a film on the surface of electrode. LDH prepared by coprecipitation and subsequent hydrothermal reaction. The CME has a good