

Linear sweep anodic stripping voltammetry: Determination of Chromium (VI) using synthesized gold nanoparticles modified screen-printed electrode

SALAMATU ALIYU TUKUR^{a,b}, NOR AZAH YUSOF^{a,c,*} and REZA HAJIAN^{c,*}

^aDepartment of Chemistry, Faculty of Science, Universiti Putra Malaysia,

43400 UPM, Serdang, Selangor, Malaysia

^bDepartment of Chemistry, Faculty of Science, Kaduna State University, Kaduna, Nigeria ^cInstitute of Advanced Technology, Universiti Putra Malaysia, Serdang, Selangor 43400, Malaysia

e-mail: azahy@upm.edu.my; rezahajian@upm.edu.my

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Abstract. A highly sensitive electrochemical sensor has been constructed for determination of Cr(VI) with the lowest limit of detection (LOD) reported to date using gold nanoparticles (AuNPs) modified screen-printed electrode (SPE). The modification of SPE by casting pure AuNPs increases the sensitivity for detection of Cr(VI) ion using anodic stripping voltammetry. Cr(VI) ions are reduced to chromium metal on SPE-AuNPs by applying deposition potential of -1.1 V for 180 s. Afterwards, the oxidation peak current of chromium is obtained by linear sweep voltammetry in the range of -1.0 V to 0.2 V. Under the optimized conditions (HClO₄, 0.06 mol L⁻¹; deposition potential, -1.1 V; deposition time, 180s; scan rate, 0.1 V s⁻¹), the limit of detection (LOD) was 1.6 pg mL⁻¹. The fabricated electrode was successfully used for detection of Cr(VI) in tap and seawater.

Keywords. Gold nanoparticles; screen-printed electrode; Cr (VI); anodic stripping voltammetry; water analysis.

1. Introduction

Hexavalent chromium is a severe environmental pollutant having carcinogenic effects in humans and animals.¹ Cr(VI) causes skin rashes and affects most of the body organs which can lead to death. Chromium is applied in industries like dyeing, electroplating, tanning, and water cooling towers. The primary source of chromium as a heavy metal pollutant is from industrial waste, which is released to rivers and air dust.^{2,3}

Chromium (VI) ion has been estimated by various methods such as laser-induced plasma spectroscopy,⁴ inductively coupled plasma mass spectroscopy/optical emission spectroscopy (ICP-MS/OES),⁵ electro-thermal atomization atomic absorption spectroscopy (ET-AAS)⁶ and electrochemical methods.^{7–10} Some of these methods are expensive and not suitable for on-site analysis. Electrochemical methods are characterized by simplicity, high sensitivity, good stability, low-cost instrumentation, small dimensions and on-site monitoring.¹¹ Electrochemical techniques especially stripping voltammetry has proven to be an excellent technique for heavy metal

detection, owing to its high sensitivity for ultra-trace analysis.

Screen-printed electrodes (SPEs) are planar electrodes that consist of plastic substrates which can be coated with layers of conducting materials and insulating inks at a controlled thickness. Since 1950, screen printing techniques have been employed in the electronics industry for fabricating printed circuit boards. The invention of SPEs with its ideal characteristics such as easy accessibility, portability, inexpensiveness, in-situ analysis and reduced sample volume represent an attractive electrochemical sensing strip for the detection of several chemical species.^{12,13} The versatile nature of SPEs has made possibile to modify them with different materials such as nanomaterials, enzymes, polymers and complexing agents.^{12,14,15} Metallic nanoparticles have received great interest in electrode modification due to their unique properties such as higher surface area, increased catalytic ability, higher signal-to-noise ratio and lower detection limit.¹⁶⁻¹⁸ Gold nanoparticles possess some good properties, such as quantized charging/ discharging, conductivity and catalytic and photocatalytic activity.¹⁹⁻²² Renedo reported the use of gold nanoparticles (AuNPs) on carbon SPE. AuNPs was deposited on to a carbon working electrode by electrodeposition

^{*}For correspondence

and Cr(VI) was detected using square wave voltammetry with a detection limit of 4.0×10^{-7} mol L⁻¹.¹⁶ Kachoosangi and Compton reported reduction of Cr(VI) in acidic medium by using gold film on carbon composite electrode for Cr(VI) detection by linear sweep voltammetry and achieved a detection limit (LOD) of 4.4 μ g L⁻¹.²³

Although there are some reports on the determination of Cr(VI) ion based on voltammetric techniques with detection limits at ppb level, there is still a need for the development of a method that is superior in accuracy, precision and speed at the levels commonly encountered in different natural samples. The methods mentioned above surmounted these problems; however, some of these methods do not have sufficiently low detection limits.

The aim of this work is to determine Cr(VI) in water resources by anodic stripping voltammetry using SPE-AuNPs modified electrode to enhance sensitivity. In this research, we fabricated an electrochemical sensor based on gold nanoparticles modified SPE for ultratrace determination of Cr(VI) ion (LOD 1.6 pg mL⁻¹) in water. This report is a combination of using acidic medium reported by Kachoosangi²³ and also dropcasting pure synthesized AuNPs onto the working electrode for improving sensitivity and low memory loss. Cr(VI) is reduced on SPE-AuNPs to form chromium metal followed by oxidation using linear sweep anodic stripping voltammetry.

2. Experimental

2.1 Materials and chemicals

All chemicals used in this study were of analytical grade. The stock solutions of Cr(VI) were prepared from $K_2Cr_2O_7(BDH \text{ Chemicals})$. Chloroauric acid (HAuCl₄) and sodium citrate dihydrate were from Sigma (USA). Perchloric acid was from Merck (Germany) and was used as the supporting electrolyte for Cr(VI) determination. Deionized water was used in all the experiments.

Screen-printed carbon electrodes based on carbon were supplied from Metrohm, Switzerland, with general dimensions of $3.4 \times 1.0 \times 0.05$ cm. The diameter of working electrode was 4.0 mm, counter electrode was made of carbon, and the reference electrode was a silver strip.

2.2 Instrumentation

Morphology of the modified electrode together with the elemental spectra of SPE-AuNPs were studied using

field emission scanning electron microscopy (FESEM) coupled with energy dispersive X-ray spectroscopy (EDS), Jeol JSM 7600F. The EDS gave the elemental spectra of AuNPs/SPE. Linear sweep voltammetry measurements were performed using a PalmSens potentiostat coupled with a computer and a SPE consisting of three different strips of carbon working electrode, carbon counter electrode, and silver reference electrode.

2.3 Modification of electrode

AuNPs was synthesized under reflux, 50 mL of HAuCl₄ (0.04 % W/V) was prepared and heated to boiling under continuous stirring, then 5 mL of 40 mmol L⁻¹ sodium citrate dehydrate, was added dropwise. The solution was further refluxed for 15 min until a stable deep red colour was observed.²⁴ SPE was modified by casting 5.0 μ L of AuNPs solution and allowed to dry under room temperature, followed by rinsing with distilled water to remove unattached AuNPs.

2.4 General procedure

An aliquot of a solution containing Cr(VI) was diluted to an appropriate concentration before commencing linear sweep voltammetry. Before each measurement, SPE-AuNPs was connected to a USB cable of potentiostat and 100 μ l of blank solution was dropped on working electrode strip, and linear sweep voltammetry was swept in the range of -1.0 to 0.2 V after deposition potential of -1.1 V for 180 s on the surface of the modified electrode. The quantitative determination of Cr(VI) ion was achieved by measuring the oxidation peak current after background subtraction at 0.05 V.

3. Results and Discussion

3.1 Characterization of modified electrode

The morphology of unmodified and modified electrodes was studied by FESEM to confirm the attachment of AuNPs onto the surface of working electrode. Figure 1 shows the images of unmodified SPE (a) and modified SPE with AuNPs (b); here AuNPs can be seen as bright white deposited points on the surface. The modified SPE shows a wide dispersion of AuNPs on the surface, which is supposed to be spherical in shape also reported by Bernalte *et al.*²⁵ Figure S1 gives the elemental spectra of AuNPs/SPE taken at different distribution, spectrum 1 (spectra 2–5 not shown) displayed two sharp peaks for AuNPs. The EDS further confirms the presence of AuNPs and it can be seen that AuNPs were widely distributed.

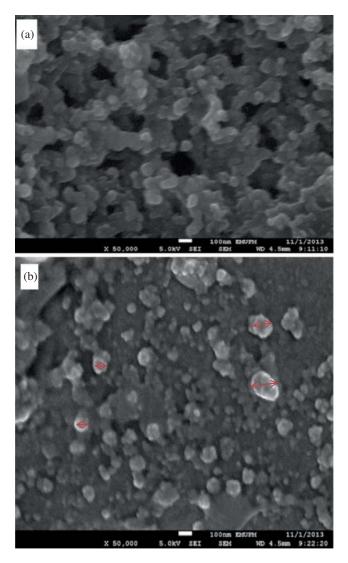


Figure 1. FESEM images for (A) bare SPE and (B) AuNPs/ SPE, AuNPs; shown as bright dots on the surface of SPE.

The effective surface area is important to enhance the sensitivity of the modified electrode due to more analyte deposition on the surface during accumulation time. Many publications had focused on a very important aspect in electrochemical studies with solid electrodes: the determination of real surface area. One of the methods pointed out by Trasatti and Petrii²⁶ is cyclic voltammetry to estimate the real surface area of working electrodes. For solid electrodes showing a welldefined double-layer region, it is possible to estimate a pseudo-capacitance through the dependence of the capacitive current with the sweep rate. In this case, the surface area can be calculated from following the equation:²⁷

$$C.A_s = \frac{\Delta i}{v} \tag{1}$$

where, C is the pseudo-capacitance of the double layer, Δi is the current variation with the sweep rate (ν) and A_s is the real surface area. Figure 2 shows cyclic voltammograms for different sweep rates on bare SPE and AuNPs/SPE in contact with a 0.06 M HClO₄ aqueous solution, in a potential range where no faradaic process occurs (-0.2 to -0.8 V). To calculate the total pseudo-capacitance of this interface following the equation 1, the cathodic currents in the range of non-faradic process were divided into one of the sweep rates (0.06 $V s^{-1}$) and the average was estimated as total pseudocapacitance. Then, the value of the cathodic current at -0.3 V was plotted against the sweep rate as shown in figure 3. The straight lines presented in figure 3 give evidence of the capacitive behaviour of the interface. From the slope of straight line, real surface area for bare SPE and SPE after modification with AuNPs was calculated as 0.306 and 1.015 cm², respectively. This finding shows that AuNPs obviously enhances the effective surface area about 4 fold).

3.2 Optimization of Parameters

Figure 4 shows linear sweep voltammograms for a solution containing 20.0 ng mL⁻¹ of Cr(VI) on SPE before and after modification with AuNPs. Cr(VI) shows a

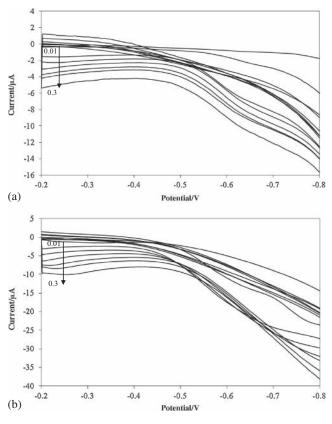


Figure 2. Cyclic voltammograms for bare SPE (A) and AuNPs/SPE (B) in 0.06 M HClO₄ aqueous solution in a non-faradaic potential range at the following scan rates: 0.02, 0.03, 0.06, 0.1, 0.15, 0.2 and 0.3 V s⁻¹.

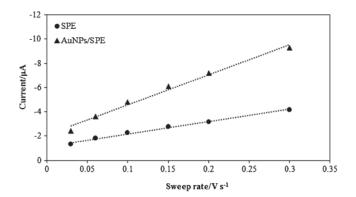


Figure 3. Anodic current at -0.3 V as a function of scan rate for the voltammograms of Figure 2

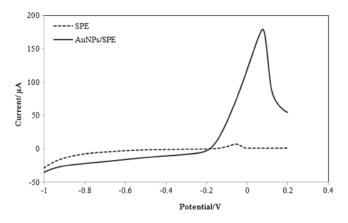


Figure 4. Linear sweep anodic stripping voltammetry of Cr(VI) ion on the surface of SPE and SPE-AuNPs modified electrode. Conditions: Cr(VI), 20 μ g L⁻¹, deposition potential, -0.8 V, deposition time, 60 s, scan rate, 0.06 V s⁻¹.

weak oxidation peak at bare SPE at around -0.05 V, due to the low surface area. While, there was a welldefined oxidation peak on SPE after modification with AuNPs in 0.1 mol L⁻¹ HClO₄ solution. The peak current was enhanced significantly (~27 folds) at lower overvoltage. The presence of AuNPs on SPE increases the microscopic surface area and also enhances the rate of electron transfer due to catalytic effect. In the following, the effects of some parameters on the sensitivity of electrochemical sensor have been studied.

3.2a The effect of supporting electrolyte: The main anionic species present for the reduction of dichromate in acidic medium (pH<4.0) is HCrO₄,^{23,28,29} while in a weakly acidic medium like perchloric acid, all the species are observed except CrO_4^- , which is negligible. Herein, perchloric acid was chosen as the supporting electrolyte due to its higher sensitivity and lower background current towards detection of Cr (VI) ion. Afterwards, the concentration of HClO₄ was optimized in the concentration range of 0.05 mol L⁻¹ to 0.2 mol

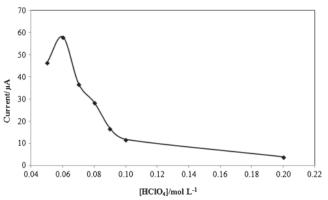


Figure 5. Effect of HClO₄ concentration on the anodic peak current of Cr(VI) using AuNPs/SPE. Conditions: Cr(VI), 7.0 μ g L⁻¹, deposition time, 120 s, deposition potential, -0.80 V, scan rate, 0.06 V s⁻¹.

L⁻¹. It was observed that Cr(VI) was easily reduced at the HClO₄ concentration of 0.06 mol L^{-1} , as it gave the highest peak current (figure 5); therefore, the concentration of 0.06 mol L^{-1} was chosen as the optimum concentration of HClO₄ for the rest of studies. Based on the information mentioned earlier and the fact that the optimum operating pH is highly acidic (pH <2.0), the following reduction reaction is tentatively suggested for reduction of chromium (VI) upon applying deposition potential at -0.8 V for 60 s. The oxidation peak in stripping linear sweep voltammetry is due to the oxidation of chromium metal to Cr(III) in the potential of 0.08 V. The extra oxidation of Cr(III) to Cr(VI) may be at potentials more positive than 0.4 V as its reduction potential (Cr(VI) to Cr(III)) is around 0.35 V. This mechanism supported also by Kachoosangi et al.23

Deposition step :
$$HCrO_4^- + H^+ \leftrightarrow H_2CrO_4$$

 $\xrightarrow{+e} CrO_3^- + H_2O \xrightarrow{6H^+, 3e} Cr^{3+}$
 $+ 3H_2O \xrightarrow{3e} Cr(m)$
Stripping step : $Cr(m) + 6H^+ + \frac{3}{2}O_2 \xrightarrow{-3e} Cr^{3+} + 3H_2O$

3.2b *Deposition potential*: The effect of deposition potential was studied in the range of -0.5 V -to 1.2 V. Figure 6 shows that the oxidation peak current increases up to deposition potential of -1.1 V. Therefore, deposition potential of -1.1 V was selected as the optimum value due to the hydrogen evaporation at more negative potentials, which can damage the surface of the modified electrode.

3.2c *Deposition time*: Deposition time is an important parameter in stripping voltammetry that has

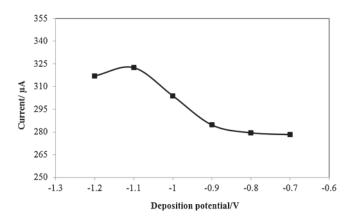


Figure 6. Effect of deposition potential on the peak current of Cr(VI) using AuNPs/SPE. Conditions: Cr(VI), 7.0 μ g L⁻¹, HClO₄, 0.06 mol L⁻¹, deposition time, 120 s, scan rate, 0.06 V s⁻¹.

influence on the sensitivity. The most important aspect of this parameter is that the deposition time leads to more accumulation of the analyte on the surface of the electrode. The effect of deposition time on the anodic stripping peak current of Cr(VI) was studied under the optimized conditions described before. Figure 7 shows that, by increasing deposition time from 20 s to 130 s, the sensitivity increases sharply, and at more times (>130 s), peak current tends to level off due to the saturation of electrode. Therefore, deposition time of 180 s was selected to achieve higher sensitivity.

The effect of potential scan rate on the anodic peak current of Cr(VI) was also studied under the optimized conditions. Increasing scan rate leads to increasing the anodic peak current from 0.01 V s⁻¹ to 0.15 V s⁻¹ with the equation of $(Ip(\mu A) = 1714.5\vartheta^{0.5} - 87.457(R^2 = 0.9986))$. The peak current is proportional to the root of scan rate based on the adsorption process during the faradic reaction.^{30,31}

3.3 *Chronoamperometry study*

In chronoamperometry studies, we determined the diffusion coefficient of Cr(VI) ion in solution upon reduction on the surface of SPE/AuNPs during deposition time at deposition potential of -1.1 V. The current for an electrochemical reaction under mass transfer control of electro-active compounds with the diffusion coefficient of D is described by the Cottrell equation:^{18,32}

$$I(t) = (nFAD^{\frac{1}{2}}C_0^*)/(\pi t)^{1/2}$$
(2)

where $I_{(t)}$ is current (A), *D* is the diffusion coefficient (cm² s⁻¹), C_{o}^{*} is the bulk concentration of analyte (mol cm⁻³) and *A* is the electrode surface area (cm²). The plot of *I* versus $t^{-1/2}$ is linear and the value of "D" can

be determined from the slope of line equation. Based on the Chronoamperometry data at 50 ng ml⁻¹ Cr(VI) under optimum parameters, the Cottrell plot was linear as follows:

$$I(t) = 7 \times 10^{-5} t^{-1/2} - 1 \times 10^{-5}$$
(3)

By considering n = 6, $A = 0.0269 \text{ cm}^2$, the value of D was estimated as $5.45 \times 10^{-5} \text{ cm}^2 \text{ s}^{-1}$. The small value of D shows that Cr(VI) cannot diffuse enough on the surface of SPE-AuNPs under diffusion mass transfer. Accordingly, the solution needs to be stirred during deposition time to enhance mass transfer by convection. It has been shown that in stripping voltammetry technique utilizing SPEs; deposition step can be performed successfully without stirring in drop-scale sample volumes.³³

3.4 Method validation

Validation of an analytical method is a procedure that is established to analyse the characteristics of a proposed method for extended analytical applications. It was examined via evaluation of the linear dynamic range, limit of detection (LOD), repeatability, precision and selectivity. Under the optimum conditions, calibration graph for the determination of Cr(VI) was prepared using linear sweep stripping voltammogram at different chromium (VI) concentrations. The calibration plot was linear in two different ranges $(0.7-3.5 \text{ ng mL}^{-1} \text{ and } 3.5-$ 35 ng mL⁻¹) with the regression equations of i_p (μ A) = 46.522 C (μ g L⁻¹) – 0.021 (R=0.9518) and i_p (μ A) $= 3.803 \text{ C} (\mu \text{g L}^{-1}) + 0.152 (\text{R}=0.9936)$ respectively. There was a slide shift in peak potential to more positive potentials as concentration of Cr (VI) increased due to the increase in the thickness of chromium metal.^{18,28} The limit of detection (defined as the amount of analyte that increases the response to $3S_b$ (S_b is the standard deviation of blank solution)),³⁴ was 1.6 pg mL⁻¹. The relative standard deviation (n = 5) for 2.0 μ g L⁻¹ of Cr (VI) was 4.5%.

3.5 Interference Study

An attractive feature of an analytical procedure is its relative freedom from interferences. Possible interference by other metal ions in the determination of chromium (VI) was investigated by the addition of some interfering ions to a solution containing 3.5 μ g L⁻¹ of chromium under optimized conditions. The tolerance limit was defined as a concentration of ion that gives an error of 10.0% or less in the determination of 3.5 μ g L⁻¹ of chromium. The result in table 1 shows that Mg(II), Hg(II), SO₄²⁻, SO₃²⁻, Cu(II) and Cd(II) are not interfering ions for determination of Cr (VI) at concentrations up to 10 times (w/w). There was no serious interference with Fe(III) and Al(III) up to 5 times (w/w) due to the non-electro-activity on the surface of modified electrode.

3.6 Real sample Analysis

To evaluate the accuracy of the proposed method for determination of Cr(VI) in real samples, the utility of

Table 1. Relative (%) change of the analytical signal of Cr(VI) in the presence of various interferences. Cr(VI) concentration, $3.5 \ \mu g \ L^{-1}$.

Foreign ions	Concentration $(\mu g L^{-1})$	Peak current change (%)	
Mg (II)	100	-2.35	
SO_{4}^{2-}	100	-3.33	
SO_3^{2-}	100	+1.57	
Hg (II)	100	+4.36	
Cu (II)	50	+5.39	
Fe (III)	50	+11.83	
Al (III)	50	+10.57	

Table 2. Determination of Cr(VI) in water samples using the fabricated electrochemical sensor (AuNPs/SPE). The results are compared with ICP–MS.

Sample	Added $(\mu g L^{-1})$	Found $(\mu g L^{-1})$	Recovery (%)	$\frac{\text{ICP-MS}}{(\mu \text{g } \text{L}^{-1})}$
Tap Water	4.0	$\begin{array}{c} 0.9 \pm \ 0.02 \\ 4.6 \pm \ 0.10 \end{array}$	92.5	<dl* 4.0± 0.07</dl*
Seawater**	4.0	$\begin{array}{c} 0.108 {\pm} \; 0.01 \\ 4.31 {\pm} \; 0.01 \end{array}$	105.0	$\begin{array}{c} 0.09 \pm 0.004 \\ 4.0 \pm 0.018 \end{array}$

* Detection limit

** Lumut, Perak, Malaysia

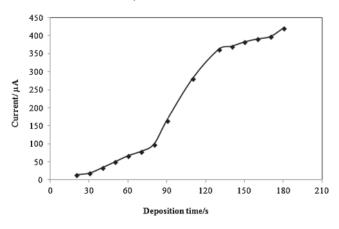


Figure 7. Effect of deposition time on the peak current of Cr(VI) using AuNPs/SPE. Conditions: Cr(VI), 7 μ g L⁻¹, HClO₄, 0.06 mol L⁻¹, deposition potential, -1.1 V, scan rate, 0.06 V s⁻¹.

the developed sensor for ultra-trace determination of Cr(VI) was investigated in tap and sea water (table 2). The data obtained for samples spiked with Cr (VI) showed good recoveries, indicating the reliability of fabricated sensor for Cr(VI) analysis in water resources. All results compared with inductively coupled plasmamass spectrometry (ICP-MS) as the standard method for Cr(VI) analysis.

4. Conclusion

In this work, an electrochemical sensor has been constructed based on SPE modified with gold nanoparticles for the determination of Cr(VI) ion in water samples. The coupling of anodic striping voltammetry with SPE-modified nanostructures enhances sensitivity and reduces sample volumes to microliter scale. This research shows that, anodic stripping voltammetry analysis of chromium (VI) ion using SPE-AuNPs is a reliable method for determination of ultra-trace amounts of chromium in water samples. The above system offers a practical method for trace determination of chromium, especially with its advantages of high sensitivity, lower detection limit (1.6 pg ml⁻¹), linear dynamic range $(0.7-35.0 \ \mu g \ L^{-1})$, high selectivity, low sample volumes, ease of fabrication, and speed compared to previous reports.

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Supplementary Information

Figure S1 (A) EDS image of AuNPs/SPE and (B) EDS spectrum from point 1 focused on AuNPs bright dot are available at www.ias.ac.in/chemsci

References

- 1. Stern A H 2010 Environ. Res. 110 798
- Wu T, Liu C, Tan K J, Hu P P and Huang C Z 2010 Anal. Bioanal. Chem. 397 1273
- 3. Han Z Q, Qi L, Shen G Y, Liu W and Chen Y 2007 *Anal. Chem.* **79** 5862
- Panne U U, Neuhauser R E, Theisen M, Fink H and Niessner R 2001 Spectrochim. Acta B 56 839
- Pereira J S F, Moraes D P, Antes F G, Diehl L O, Santos M F P, Guimaraes R C L, Fonseca T C O, Dressler V L and Flores E M M 2010 *Microchem. J.* 96 4

- 6. Chaguaramos L 2012 Talanta 97 505
- 7. Korolczuk M 2000 Anal. Chim. Acta. 414 165
- 8. Liu C, He C, Xie T and Yang J 2015 *Fuller. Nanotub. Car. N.* **23** 125
- 9. Korolczuk M and Grabarczyk M 1999 Anal. Chim. Acta. 387 97
- 10. Kumar Jena B and Retna Raj C 2008 Talanta 76 161
- Dajas F, Rivera F, Blasina F, Arredondo F, Echeverry C, Lafon L, Morquio A and Heizen H 2003 *Neurotox. Res.* 5 425
- 12. Yusof N A, Daud N, Zareena S, Saat M and Tee T W 2012 Int. J. Electrochem. Sci. 7 10358
- 13. Khun N W and Liu E 2009 Electrochim. Acta 54 2890
- Rahman N A, Yusof N A, Amirah N, Maamor M, Mariam S and Noor M 2012 Int. J. Electrochem. Sci. 7 186
- 15. Daud N, Yusof N A and Tee T W 2011 Int. J. Electrochem. Sci. 6 2798
- Costa-Garcia A, Martinez-paredes G and Bego M 2009 Electrochim. Acta 54 4801
- Dominguez-Renedo O, Ruiz-Espelt L, Garcia-Astorgano N and Julia Arcos-Martinez M 2008 *Talanta* 76 854
- 18. Xing S, Xu H, Chen J, Shi G and Jin L 2011 J. Electroanal. Chem. 652 60
- 19. Chen S and Murray R W 1999 J. Phys. Chem. B 103 9996

- 20. Li J, Yamada Y, Murakoshi K and Nakato Y 2001 *Chem. Commun.* **21** 2170
- 21. Haruta M 1997 Catal. Today 36 153
- 22. Subramanian V, Wolf E E and Kamat P V 2003 *J. Phys. Chem. B* **107** 7479
- 23. Kachoosangi R T and Compton R G 2013 Sensor Actuator B: Chem 178 555
- 24. Hajian R, Yusof N R, Faragi T and Shams N 2014 *Plos One* **9** e96686
- 25. Bernalte E, Marin Sanchez C and Pinilla Gil E 2012 Sensor Actuator B 161 669
- 26. Trasatti S and Petrii O A 1991 Pure Appl. Chem. 63 711
- Salkind A J and Yeager E 1972 In *Techniques of Electrochemistry* Vol. 1 (New York: Wiley Interscience) p. 293
- 28. Bergamini M F, dos Santos D P and Zanoni M V B 2007 Sensor Actuator B **123** 902
- 29. Pladziewicz J R and Espenson J H 1971 *Inorg. Chem.* 10 634
- 30. Ensafi A A and Hajian R 2005 Electroanalysis 18 579
- 31. Somerset V, Leaner J, Mason R, Iwuoha E and Morrin A 2010 *Electrochim. Acta* **55** 4240
- 32. Lingane J J 1941 Chem. Revs. 29 1
- Jin-Ming J, Yan-Yan L, Ye-Lei Z, Xi-Shan G and Qiang C 2013 Sensors 13 13063
- 34. Christian G D 2004 Analytical Chemistry 6th edition (John Wiley) p. 113