

Magnesium layered hydroxide-3-(4-methoxyphenyl) propionate modified single-walled carbon nanotubes as sensor for simultaneous determination of Bisphenol A and Uric Acid

Rahadian Zainul^{1,*}, Norhayati Hashim², Siti Nur Akmar Mohd Yazid², Sharifah Norain Mohd Sharif², Mohamad Syahrizal Ahmad², Mohamad Idris Saidin², Suyanta³, M.M.C. Sobry² and Illyas Md Isa^{2,*}

¹ Department of Chemistry, Faculty of Mathematics and Natural Science, Universitas Negeri Padang, West Sumatera, 25173, Indonesia

² Nanotechnology Research Centre, Faculty of Science and Mathematics, Universiti Pendidikan Sultan Idris, 35900 Tanjong Malim, Perak, Malaysia;

³ Department of Chemistry Education, Faculty of Mathematics and Natural Science, Yogyakarta State University, Indonesia

*E-mail: rahadianzmsiphd@fmipa.unp.ac.id; illyas@fsmt.upsi.edu.my

Received: 12 May 2021 / Accepted: 27 June 2021 / Published: 10 August 2021

A facile electrochemical sensor based on single-walled carbon nanotubes/magnesium layered hydroxide-3-(4-methoxyphenyl) propionate nanocomposites paste electrode (MWCNT/MLH-MPP) for determination of bisphenol A (BPA) and uric acid (UA) was constructed in this study. According to experiments, several parameters affecting the electroanalytical responses of the prepared electrode were optimised, such as amount of modifier, square wave voltammetry parameters, and pH effect. Under optimum experimental conditions, electrochemical current of BPA and UA oxidation were linearly proportional to the concentrations from 3.0×10^{-7} M to 1.0×10^{-4} M and 1.0×10^{-7} M to 1.0×10^{-4} M for BPA and UA respectively with the both limit of detection of 5.0×10^{-8} M. The small percentage of relative peak currents changes with added some interference indicated that the modified electrode can be used for the determination of BPA and UA in real samples such as baby teether and urine.

Keywords: single-walled carbon nanotube; nanocomposite; bisphenol A; uric acid; electrochemical sensor

1. INTRODUCTION

Bisphenol A (BPA) [4, 4'-dihydroxy-2, 2-diphenylpropane] (Fig. 1), is a major industrial chemical used primarily to make polycarbonate plastic, epoxy resins and baby bottles. In the process of production, addition of BPA makes the plastic products be more transparent, durable, and shockproof

[1,2]. BPA has attracted extensive attention due to the widespread use of these materials has caused a serious of environmental problems as it can migrate from waste plastics into the water, and thus damage the health of people. Uric acid (UA) is the primary end product of purine metabolism, which is present in biological fluids including urine and blood. UA can displays keto-enol tautomerism (Fig. 1). The normal concentration of UA in human body is in the range of 4.0 to 8.0 mg/dL (from 0.237 to 0.476 mM) [3] and a higher level of UA than that range leads to a possibility of causing gout, kidney stones, and uremia [4]. Therefore, it is crucial to detect BPA and UA selectively and simply in the diagnosis and treatment of diseases.

Currently, various analytical techniques for determination of UA and BPA have been established mainly including high-performance liquid chromatography [5,6], gas chromatography-mass spectrometry [7,8], and capillary electrophoresis [9,10]. However, those require bulky equipment, sample pre-treatment, harmful solvent, skill, time, and cost. In particular, electrochemical technique has attracted much attention owing to its ease of operation, high sensitivity, good selectivity and excellent convenience, as well as instant analysis [11,12]. However, to measure UA, some biological samples can cause interference with these assays such as ascorbic acid and dopamine, due to the fact that their oxidation potentials are close to that of UA [13]. Meanwhile, a direct detection of BPA using a traditional electrochemical sensor is usually very poor. A relatively high potential is required for the oxidation of phenolic compounds, hence, increase of the background current and a lowered sensitivity [14]. Thus, this led to the development of electrochemical methods that can selectively and sensitively measure the UA and BPA.

Carbon nanotubes (CNTs) has attracted enormous attention as an excellent modifier and been employed in preparing electrochemical sensors due to their unique structural and electronic properties (such as large surface area, high electrical conductivity and so on). Solubility of CNTs in solvents normally is very low so that it's suitable to develop as electrochemical transducers, i.e paste electrodes by adding with certain amount of electroactive compound or material and mineral oil or epoxy resin as a binder. Several electroanalysis that use modified carbon nanotubes paste electrodes have been described, and different compounds have been added as active modifiers [15–17].

Magnesium layered hydroxide-3-(4-methoxyphenyl) propionate (MLH-MPP) is a kind of two-dimensional layered metal hydroxide (LMH) material, which closely related to anionic clay. The general formula to described LMH is $M^{2+}(OH)_{2-x}(A^{n-})_{x/n} \cdot yH_2O$, where M^{2+} is the metallic cation such as Co^{2+} , Mg^{2+} , Ni^{2+} , and Zn^{2+} . A is a counter ion with a negative charge [18,19]. LMHs have a special layered structure and variable chemical compositions that can be readily tuned. So that, LMHs have been widely applied in the field of biomedicine, conversion, electrochemical energy storage, and photoluminescence [20–22]. However, less attention has been paid to the use of LMH as an electrochemical sensor.

Following this, we decided to explore a novel of composite modified carbon paste electrode for electrochemical simultaneous BPA and UA sensor using MLH-MPP and single-walled carbon nanotubes (SWCNTs).

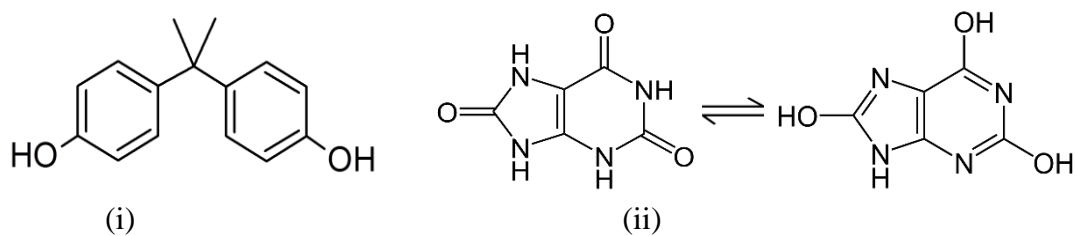


Figure 1. Structure of bisphenol A (i) and uric acid (ii).

2. EXPERIMENTAL

2.1. Chemical and reagents

BPA and UA were obtained from Sigma-Aldrich (USA). The SWCNT from was obtained from Timesnano (China). The MLH-MPP was synthesised according to previous reported [23]. The phosphate buffer solutions (PBS) were prepared from K_2HPO_4 and KH_2PO_4 (Merck), adjusting the pH as required with 0.5 mol L^{-1} NaOH and HCl.

2.2. Instruments

The electrochemical cell was constituted by a three-electrode system using Gamry Potentiostat Series-G750 (USA). The Ag/AgCl was used as the reference electrode, a platinum wire was functioned as the auxiliary electrode and bare or modified SWCNT was used as working electrode. The pH measurements were firstly calibrated using standard pH buffer solutions, then adjusted with Thermo Scientific Orion 720A Benchtop pH meter (Thermo Fisher Scientific Corp. USA). The surface morphology of MLH-MPP was conducted by field emission scanning electron microscopy, model Hitachi SU 8020 UHR (Japan). The electrochemical impedance spectroscopy (EIS) measurements were carried out on Potentiostat/Galvanostat Gamry Ref 3000 (USA). Finally, the real samples validation analysis were conducted by using high performance liquid chromatography (HPLC).

2.3. Preparation of the modified electrode

The modified electrode was prepared manually using mortar and pastel for mixing about 100 mg SWCNT with 5, 10, 15, and 20 mg MLH-MPP, respectively. The paraffin oil was added for about two droplets which functioned as binder and the mixture was grinded homogeneously. The paste obtained was firmly packed into Teflon tubes (id = 2.0 mm, 3.0 cm long), followed by an inserted of a copper wire at one end of the tubing to produce electrical contact with the potentiostat.

2.4. Measurement of BPA and UA

Voltammetry measurements were performed using 0.1 M PBS (pH 6) as the electrolyte in the simultaneously presence of 0.1 mM BPA and UA at range of 0 V to +0.9 V by utilizing square wave voltammetry (SWV) with frequency (120 Hz); pulse height (60 mV); step increment (6 mV). The

electrode surface was required to be smoothed with filter paper before conducting the measurement. Every experiment was performed at room temperature of $25 \pm 2^\circ\text{C}$.

2.5. Real samples analysis and validation study

Prior to study the concentration of BPA, a baby teether was soaked into a beaker containing 20.0 mL ethanol and heated for 4 hours at 50.0°C . The mixture was filtered and the solvent obtained was completely evaporated. Then, 3.0 mL ethanol was added to the residue and diluted with 50.0 mL PBS before measuring. While the urine samples were collected from two healthy person and diluted 100 times with PBS buffer (pH 6), then UA was analysed without other pre-treatment.

The validity of the sensor was compared with HPLC (Agilent 1200 Infinity Series (Germany), G4212B UV-Visible diode array detector equipped with Agilent G1367B autosampler). Reversed phase C-18 column used was Zorbax Eclipse-AAA 4.6×150 mm, $3.5 \mu\text{m}$ particles. The mobile phase used in this study was acetonitrile/ water (v/v) at a flow-rate of 1.5 mL min^{-1} . The absorption detector was operated at 280 nm.

3. RESULTS AND DISCUSSION

3.1. Surface morphology and electrochemical impedance spectroscopy study

The morphological studies of the SWCNT/MLH-MPP were observed by scanning electron microscope (SEM) and transmission electron microscope (TEM). Fig.2A clearly shows the presence of SWCNT bundles covered with SWCNT/MLH-MPP. While TEM analysis of the hybrids in Fig. 2B shows that SWCNT is well dispersed in presence of MLH-MPP. Also TEM image shown that MLH-MPP as opaque features was covered by transparent delicate tube of SWCNT. These results clearly revealed the existence of MLH-MPP along with SWCNT.

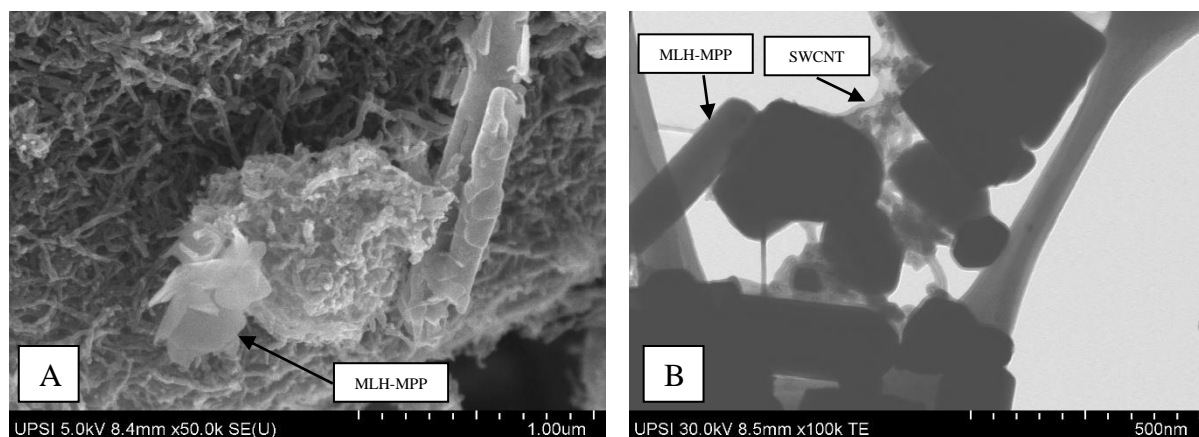


Figure 2. FESEM (A) and TEM(B) images of SWCNT/MLH-MPP.

The capability of electron transfer on the surface of the electrodes study was studied by performing the electrochemical impedance spectroscopy (EIS) analysis on 4.0 mM $K_3[Fe(CN)_6]$ solution containing 0.1 M KCl. In the Nyquist diagrams, the diameter of the semicircle at higher frequencies presents the interfacial electron transfer resistance, R_{ct} , and the linear portion at lower frequencies corresponds to the diffusion process [24]. Fig. 3 illustrates the EIS for unmodified and modified SWCNTs paste electrode. By fitting Randles equivalent electrical circuits (Fig. 3 inset) the R_{ct} values for unmodified and modified carbon paste electrodes are 10.57 k ohms and 0.795 k ohms, respectively. Obviously, the unmodified carbon paste electrode exhibits a large semicircle with high R_{ct} value indicating the lowest electron transfer rate. In contrast, a very small semicircle nearly straight-line exhibits by modified SWCNTs paste electrode indicating that the modified electrode is able to accelerate the electron transfer rate and it is controlled by diffusion. This EIS result was in accordance with the cyclic voltammetry studies which is contributed to the excellent conductivity of electrode.

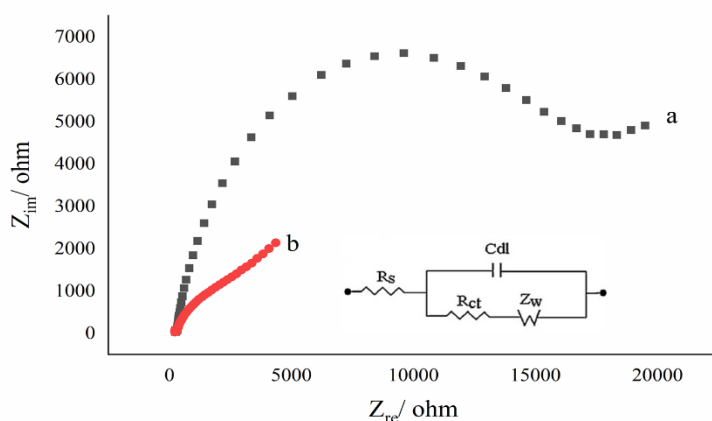


Figure 3. Nyquist plot of the (a) unmodified SWCNT and (b) SWCNT/MLH-MPP paste electrodes in 4.0 mM $K_3[Fe(CN)_6]$ in the presence of 0.1 M KCl. Inset: Randle's equivalent electrical circuit system.

3.2. Optimization of amount of modifier

Fig. 4 presents the SWV responses of unmodified and modified SWCNT with different percentage of MLH-MPP modifier in the presence of 0.1 mM BPA and UA in 0.1 M PBS. It was found that by increasing in the amount of modifier from 5 to 15 mg, the peak currents were enhanced, while further increasing in the amount of modifier to 20 mg, resulted in significant decreased of peak current. This suggests that the addition of MLH-MPP modifier into the SWCNT has affected and altered some of the physical and physiochemical properties on the electrode surface leading to the increasing of its conductivity. Somehow until certain level, the surface of the modified SWCNTs paste electrode became saturated due to the excess loading of the modifier, thus reducing the rate of electron transfer at the electrode/solution interface [25]. Therefore, in this study, 15 mg of MLH-MPP modifier was selected for preparation of SWCNT-modified electrode.

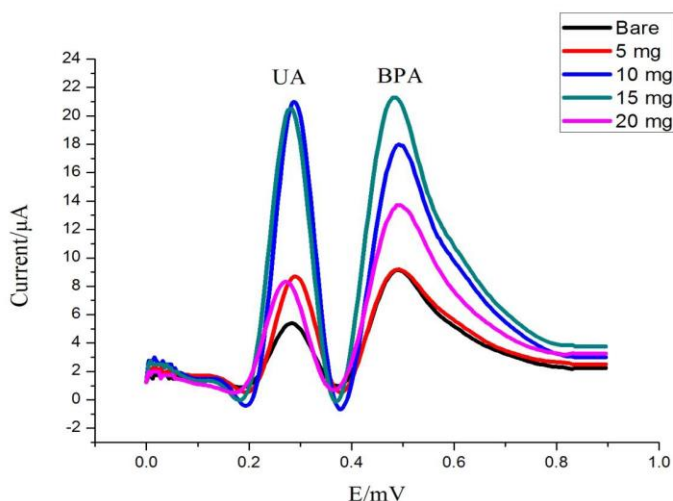


Figure 4. SWV response of unmodified and modified SWCNT with 5, 10, 15 and 20 mg MLH-MPP, respectively, in 0.1 M PBS containing 0.1 mM BPA and UA simultaneously.

3.3. Effect of SWV parameters

In this study, a few parameters such as frequency and pulse size were further investigated in coherent to a previous theoretical calculations performed by Osteryoung & Osteryoung [26] proposed that the voltammogram characteristic was linearly dependent on some parameters used. The relationship between the resulted peak currents and the parameters were presented in Figure 2. The effect of frequency was evaluated by increasing the frequency from 20 to 160 Hz. By increasing the frequency from 20 to 120 Hz resulting the increasing peak currents towards simultaneous detection of BPA and UA, but later decreasing at over 140 Hz (Fig. 5a). In addition, Fig. 5b presents the effect of pulse size parameter in the range of 10 to 60 mV. The pulse size of 60 mV was selected due to highest peak current and generate a good peak resolution.

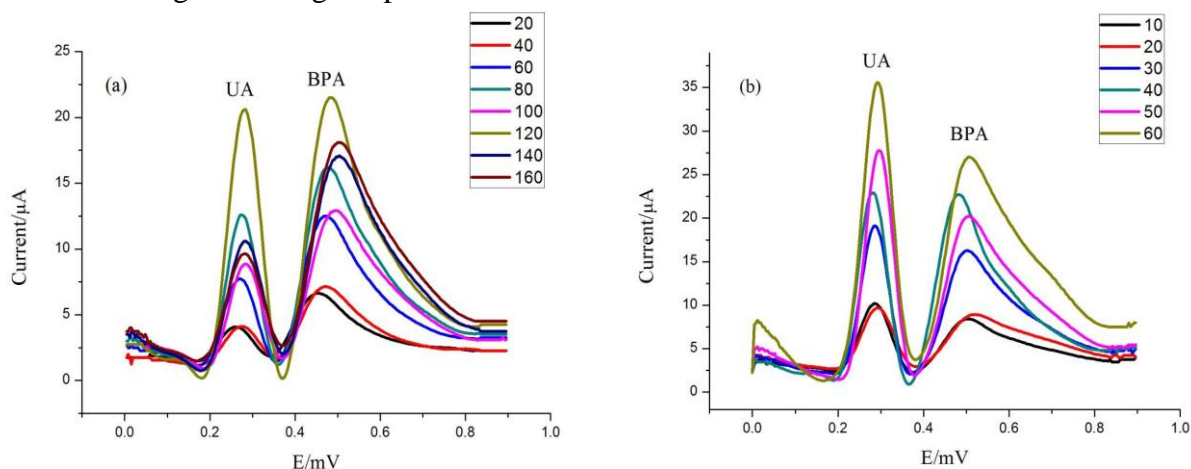


Figure 5. Influence of the SWV parameters (a) frequency, Hz (b) pulse size, mV and on signal of 0.1 mM UA and BPA.

3.4. Effect of electrolyte

The SWV peak currents were also taken into consideration based on the selection of the supporting electrolyte. In this study, the electrochemical responses of SWCNT/MLH-MPP paste electrode towards simultaneous detection of BPA and UA was also tested using different type of electrolytes, such as CH_3COONa , KCl , KNO_3 , LiCl , NaCl , and PBS. As shown in Fig. 6, the SWV peak current towards simultaneous detection of BPA and UA were achieved the best using PBS electrolyte. This can be explained by the fact that in PBS solution, the adsorption of BPA and UA is more easily towards the electrode surface as there is more efficient ability in increasing the anion and cation charge compensation, thus leads to a higher peak current. Therefore, the PBS solution has been chosen as the electrolyte for subsequent studies.

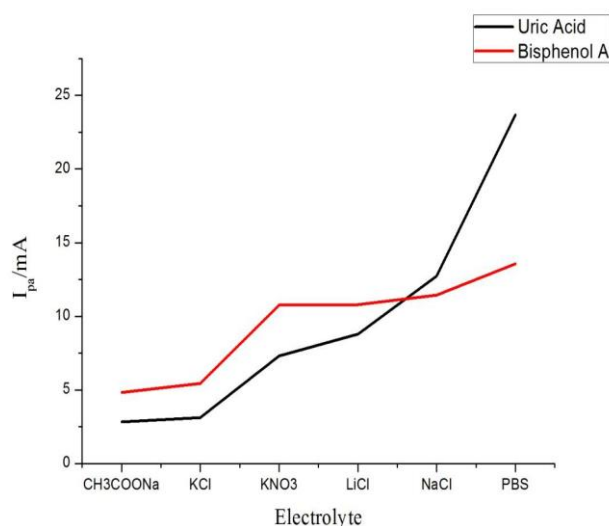


Figure 6. Effect of different types of supporting electrolyte (CH_3COONa , KCl , KNO_3 , LiCl , NaCl and PBS) towards current on the simultaneous detection of 0.1 mM BPA and UA.

3.5. Effect of pH

Fig. 7 shows the effect of pH (2 – 8) on the peak potentials of BPA and UA at the SWCNT/MLH-MPP paste electrode in 0.1 M PBS. The peak currents of BPA and UA increased with an increment in pH value from 2 to 6, and decreased when the pH was higher than 6 due to protonation of H-bonded OH in BPA and UA (enol form). Thus, PBS electrolyte with the pH value of 6 was chosen as the optimum experimental parameter in the following BPA and UA detection.

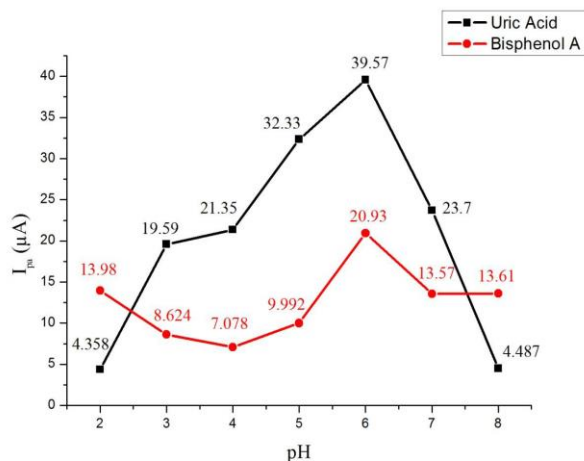


Figure 7. SWVs of the SWCNT/MLH-MPP paste electrode in the simultaneous presence of 0.1 mM BPA and UA at different pH values of 0.1 M PBS electrolyte.

3.6. Simultaneously calibration analysis

The simultaneous electroanalytical determination of BPA and UA using the SWCNT/MLH-MPP paste electrode was performed by SWV obtained for different BPA and UA concentrations are shown in Fig. 8. These peak currents presented good linear relationship with the concentration of BPA and UA. The linear regression equations were $I (\mu A) = -7.87 + 44.269 C (\mu M)$ ($R^2 = 0.9983$) for BPA and $I (\mu A) = -3.5305 + 19.663 C (\mu M)$ ($R^2 = 0.997$) for UA, respectively. The performance of the SWCNT/MLH-MPP paste electrode in determination of UA and BPA was compared to those method reported earlier (Table 1).

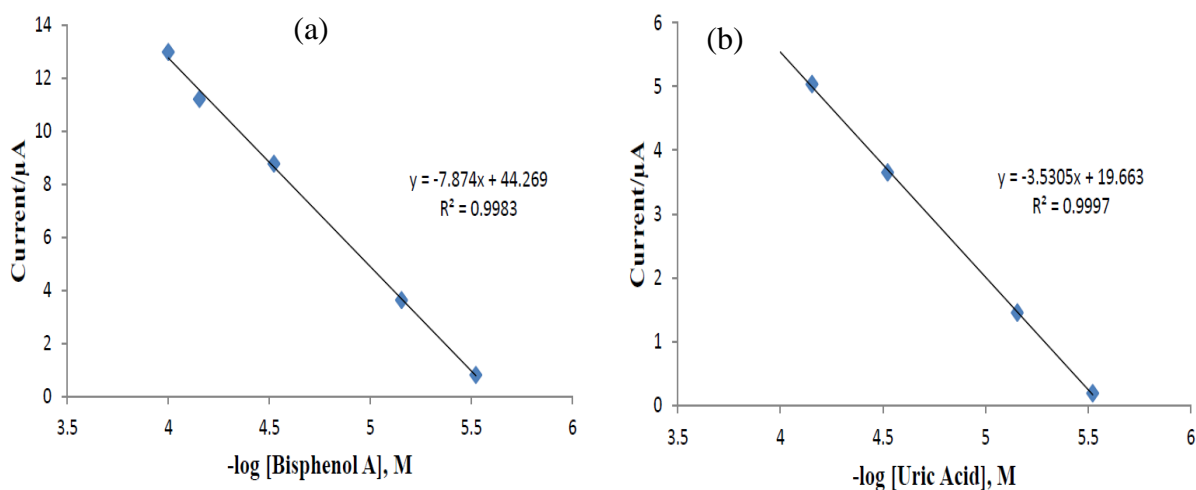


Figure 8. Calibration plots of different concentrations of (a) BPA and (b) UA using the SWCNT/MLH-MPP paste electrode.

Table 1. Comparison between the this work and some reported sensors for the determination of BPA and UA

Type of determination	Analyte	Modifier/electrode	LWR (μM)	LOD (μM)	Ref.
Individual	UA	cetyltrimethylammonium bromide /CPE	5.0 – 125.0	1.7	[27]
		5-(4-Aminophenyl)-10,15,20-triphenylporphyrin]Mn(III)/GCE	0.5 – 500.0	0.300	[28]
		gold microclusters /GCE	0.2 – 50.0	0.12	[29]
	BPA	1-aminopropyl-3-methylimidzaolium tetrafluoroborate/GCE	0.02 – 3	4.6	[30]
		Tween 20/CPE	1 – 16	0.12	[31]
		zinc phthalocyanine tetrasulfonic acid/GCE	0.05 – 4	0.02	[32]
Simultaneous	UA	Zn-Al-LDH-QM/MWCNT/CPE	0.1 – 100.0	0.05	This work
	BPA		0.3 – 100.0	0.05	

CPE – Carbon paste electrode; GCE – Glassy carbon electrode; LWR – Linear working range; LOD – Limit of detection.

3.7. Reproducibility, repeatability, stability and interference studies

The reproducibility, repeatability and stability of the sensors were taken into account in this study. The reproducibility experiment was evaluated by measuring the response of both 0.1 mM BPA and UA solution using three different electrodes which were prepared by the same procedure. Relative standard deviations (RSD) calculated were 0.88 % and 1.20 %, for BPA and UA respectively. The repeatability experiment was carried out by a series of repetitive measurements of 0.1 mM BPA and UA solutions at optimum condition. The RSD of 2.84 % and 2.73% were obtained for BPA and UA, respectively. Besides that, the electrode was stored for four weeks in order to investigate the electrode stability towards response of BPA and UA. The result shown that the response current values retained 98.65 % of the initial current for BPA, and 96.73 % of the initial current for UA.

The selectivity of the prepared electrode was investigated in the presence of potential interferences towards BPA and UA detection (Fig. 9). Under optimum condition, the signal change was less than 9% for 10-fold higher and less than 25% for 100-fold higher than that of BPA and UA, respectively.

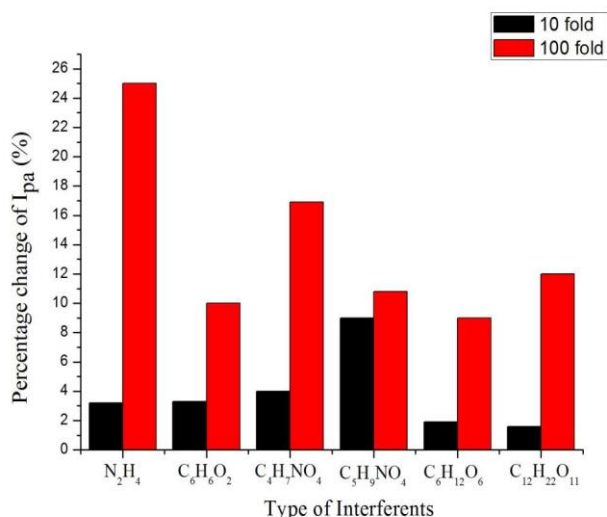


Figure 9. The effect of potential interferences in 0.1 mM BPA and UA at SWCNT/MLH-MPP paste electrode

3.8. Real sample study

The determination of BPA and UA in baby teether and urine samples was applied to test the applicability of the SWCNT/MLH-MPP paste electrode. The samples were spiked with standard BPA and UA solution at different concentrations according to standard addition method and the percentage of recoveries were calculated. Table 2 shown that recoveries of the measurement were 96.0 – 98.0 % and 98.0–100.4 % for BPA and UA respectively, indicated that the SWCNT/MLH-MPP paste electrode is appropriate for the analysis of real samples.

The baby teether and urine samples were also measured using HPLC technique for comparison purpose (Table 3). An independent *t*-test was performed at a confident level of 95 % to study the relationship between the results obtained from SWV and HPLC techniques. Since *p*-value (.198) and (.055) for baby teether and urine sample are greater than the significance level $p > .05$, the null hypothesis failed to be rejected, indicating there is no significant difference between the data obtained from SWV and HPLC techniques.

Table 2. Recoveries of BPA and UA in baby teether and urine.

Sample	Detected (μM)		Added (μM)	Found (μM)	Recovery (%)	
	BPA	UA				
Urine 1	^a ND	18.8	UA	5.0	23.5	98.7
			BPA	5.0	4.8	96.0
Urine 2	ND	20.2	UA	5.0	23.9	100.4
			BPA	5.0	4.9	98.0
Baby teether	2.67	ND	UA	5.0	4.9	98.0
			BPA	5.0	7.5	97.8

^a ND – Not detected.

Table 3. Comparison study of SWV and HPLC techniques.

Sample	Method	Mean (μM)	Std. Deviation	Sig. (2-tailed)
Urine	SWV	21.73	0.46	0.116
	HPLC	22.20	0.35	
Baby teether	SWV	19.97	0.30	0.098
	HPLC	19.56	0.38	

4. CONCLUSIONS

In conclusion, a novel electrochemical sensor for simultaneous detection of BPA and UA has been developed based on MLH-MPP-modified SWCNT paste electrode. A fixed SWCNT loadings and variety amounts of MLH-MPP modifier were tested and the best experimental conditions were 15 mg of MLH-MPP in 100 mg of SWCNT. The investigated MLH-MPP modifier appeared to be an effective electrocatalyst for the detection of BPA and UA. The sensor achieved a good linear relationship between the electrochemical current response and concentration of BPA and UA. This study made a great contribution to broadening application prospects for the development of BPA and UA sensors in the future.

ACKNOWLEDGEMENT

This research was funded by Government of the Republic of Indonesia, Rector of Universitas Negeri Padang for grant number 1412/UN35.13/LT/2020 and 1008/UN35.13/LT/2021. Also authors acknowledge the cooperation from the Universiti Pendidikan Sultan Idris, Tg. Malim, Perak, Malaysia for the success of the research.

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