Int. J. Electrochem. Sci., 15 **(2020)** 6177 – 6187, doi: 10.20964/2020.07.03

International Journal of
ELECTROCHEMICAL
SCIENCE
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Synthesis of Au Nanoparticles Decorated Carbon Nanotubes as an Electrochemical Sensor for Phenol Determination in Petrochemical Wastewater

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Received: 10 October 2019 / *Accepted:* 24 December 2019 / *Published:* 10 June 2020

Phenolic compounds are identified as pollution in wastewater due to the petroleum industry. These hazard materials lead to many illnesses such as cancer. Therefore, study on the phenolic compounds and the detection techniques are necessary. In this study, Au nanoparticles decorated carbon nanotubes (Au NPs/CNTs) electrode was applied to determine phenol. CNTs were synthesized through chemical vapor deposition technique. The Au nanoparticles were decorated on synthesized CNTs by electrochemical deposition. The structural properties of synthesized CNTs and Au NPs/CNTs were studied by FESEM, XRD, and Raman analyses. Results showed that high density of CNTs with high aspect ratio and high effective surface area were synthesized. Electrochemical properties of Au NPs/CNTs electrodes were investigated by cycle voltammetry and differential pulse voltammetry techniques. The electrochemical characteristic of Au NPs/CNTs electrode showed oxidation sharp peak in 0.57 v for phenol in pH 7. The result of cycle voltammetry study showed the stability behavior of Au NPs/CNTs electrode for phenol oxidation. Differential pulse voltammetry studied revealed that modified sensor was selective and sensitive in determination of phenolic compounds. The linear range, sensitivity, and detection limit of sensor were estimated 1-200 μM, 0.499 μA/ μM and 6 nM, respectively, which were more competitive and superior than the previous reports.

Keywords: Au nanoparticles, Carbon nanotube-based sensor; Phenolic Compounds; Petrochemical Wastewater; Cycle voltammetry; Differential pulse voltammetry

1. INTRODUCTION

Recently, concerns among scientists and politicians have increased due to the effect on wildlife and human that are exposed to chemicals in the environment, especially the aquatic environment.

Among the chemical compounds, phenolics are a major concern in this area because they tend to stay in the environment for a long time which applies a toxic effect on animals and humans [1]. Some phenolic compounds exist extremely in nature that are particularly related to the color of fruits and flowers [2]. Most well-known phenolic compounds included phenol, 4-chlorophenol, 2,4-dichlorophenol, 2,4,6-trichlorphenol. Therefore, there is a great concern worldwide to eliminate these pollutants from industrial wastewater before discharging them into aquatic ecosystems. Then, it is imperative for industries to purify their wastewater to ensure the disposal is safe for the environment.

The phenol and its derivatives represent a potential source of pollution and are usually found in wastewaters of different factories and petrochemicals [3]. Phenol pollution also has serious consequences for the environment due to its harmful effects on aquatic organisms [4, 5] including algae and aquatic spermatophytes. Various techniques, such as liquid chromatography-mass spectrometry, high-performance liquid chromatography, gas chromatography-mass spectrometry, and electrochemical methods have been considered for the detection of phenolic compounds and its derivatives [6-8]. These conventional techniques are reliable, sensitive and precise. Despite the many advantages, most of these techniques require expensive instruments, skilled technicians, and high execution time. Among these techniques, electrochemical methods such as cyclic voltammetry, and differential pulse voltammetry is fast response and low-cost sensing approach in detecting many of the phenolic compounds [9].

Currently, the use of nanostructure for modification of the electrode surface for the detection of organic and non-organic compounds was reported [10]. Among the nanostructures, carbon nanotubes with unique one-dimensional structure, mechanical properties and outstanding electrical can be used as electrode material for sensing the phenolic compounds [11]. Moreover, carbon nanotubes structures can enhance the electron transfer rate in electrochemical activities and high porosities in carbon nanotube structures can improve the sensing properties of phenol components such as fast response, detection limit, sensitivity and stability of the sensor.

Several nanostructures have been used for various purposes, but carbon nanotubes developed as the most versatile and exciting [12]. Carbon nanotube (CNTs) materials have positive effects in applied sciences which has obtained more attention because of the higher yield and the ability to improve its electrical properties [13, 14], thus making them suitable for various applications in energy storage, transistors, hybrid conductors, field emitters, thin-film coatings, photovoltaic devices, and electrochemical probes [15]. The decoration of gold nanoparticles (Au NPs) on CNTs in the preparation of nano-hybrid materials has many interesting applications in biosensors [16], gas sensors [17] and drug delivery [18] because of its high stability and sensitivity.

In this study, Au NPs/CNTs electrode was applied for the determination of phenol as a composition in wastewater of petroleum industry. Therefore, in the first step, carbon nanotubes were synthesized through chemical vapor deposition technique. In the second step, The Au nanoparticles were decorated on synthesized CNTs by electrochemical deposition. Then, the structural properties of synthesized CNTs and Au NPs/CNTs were studied by SEM, XRD, and Raman analyses. Finally, electrochemical sensing properties of carbon nanotubes electrode were investigated for sensing the phenol by using cycle voltammetry and differential pulse voltammetry techniques.

2. EXPERIMENTAL

CNTs were synthesized on a Si (100) substrate using chemical vapor deposition (CVD) method including a two-stage tube furnace. Ferrocene (5.3 wt.%) was carefully mixed in palm oil as a precursor. Then, a 5 ml mixture was positioned in an alumina boat located side-by-side in the CVD reaction furnace. Si substrates were located in the center of next furnace. Then, the reaction furnace was heated to 190 °C to vaporize precursors. The temperature was increased to 800 °C in the second furnace, before increasing the temperature to 400 °C in the first furnace. An argon gas flow of 200 sccm was injected for 40min into the inlet tube up to full consumption of the carbon source.

In order to fabricate the Au nanoparticles modified carbon nanotubes (Au NPs/CNTs), the CNTs electrode was immersed into a 5.0 mM of chloroauric acid ($HAuCl_4$) solution containing 0.1 M potassium nitrate. A constant potential of - 0.45 V vs. Ag/AgCl was applied for 2 minutes. Then, the modified electrode was washed with doubly distilled water and dried at room temperature.

The morphology of the synthesized CNTs was analyzed by scanning electron microscopy (FESEM, Gemini Zeiss Supratm 35 VP, Carl Zeiss). Crystallinity analysis of CNTs and Au NPs/CNTs was performed with Xpert Pro X-ray diffractometer with 1.5404 Å (Cu Kα) in wavelength and 40KV/30 mA in power. Raman spectroscopy (Labram HR Evolution, Germany) was used to study the vibrational and frequency modes in CNTs and Au NPs/CNTs.

Electrochemical studies were performed in the three-electrode cell which contains Ag/AgCl/(sat KCL), a Pt wire and CNTs and Au NPs/CNTs as a reference, counter electrode, and the working electrodes, respectively. Auto lab modular electrochemical system (Eco. ChemieulTecht) was used for voltammetry studies. The 0.1 M phosphate buffer solutions were provided from H_3PO_4 and NaH_2PO_4. The pH of the phosphate buffer solutions was adjusted by HCl and NaOH solution. The cyclic voltammetry analysis was carried out between 0 V and 1 V at scan rate with 20 mV/s. Differential pulse voltammetry (DPV) was used the under following conditions: 0.4–0.7 V potential range, 60 mV pulse amplitude and 10 mV/s scan rate.

3. RESULTS AND DISCUSSION

FESEM analysis can provide information on the morphological structure of nanostructure on electrode surface. Figure 1a shows the SEM image of synthesized carbon nanotubes. The high density of nanotubes with a length of about 10 µm is observed. As seen, the diameter of CNTs is approximately 80 nm. Therefore, high aspect ratio and high effective surface area of CNTs were synthesized. Moreover, high porosity surface was prepared for carbon nanostructures. These characterizations of electrode can promote the sensor properties in the determination of the analytes [19]. The grafting functionalized CNTs using gold NPs solution is then performed. Figures 1b shows SEM images of AuNPs decorated CNTs. It is clearly seen that numerous Au NPs on the surface of CNTs can be observed. They are well distributed on the whole CNTs.

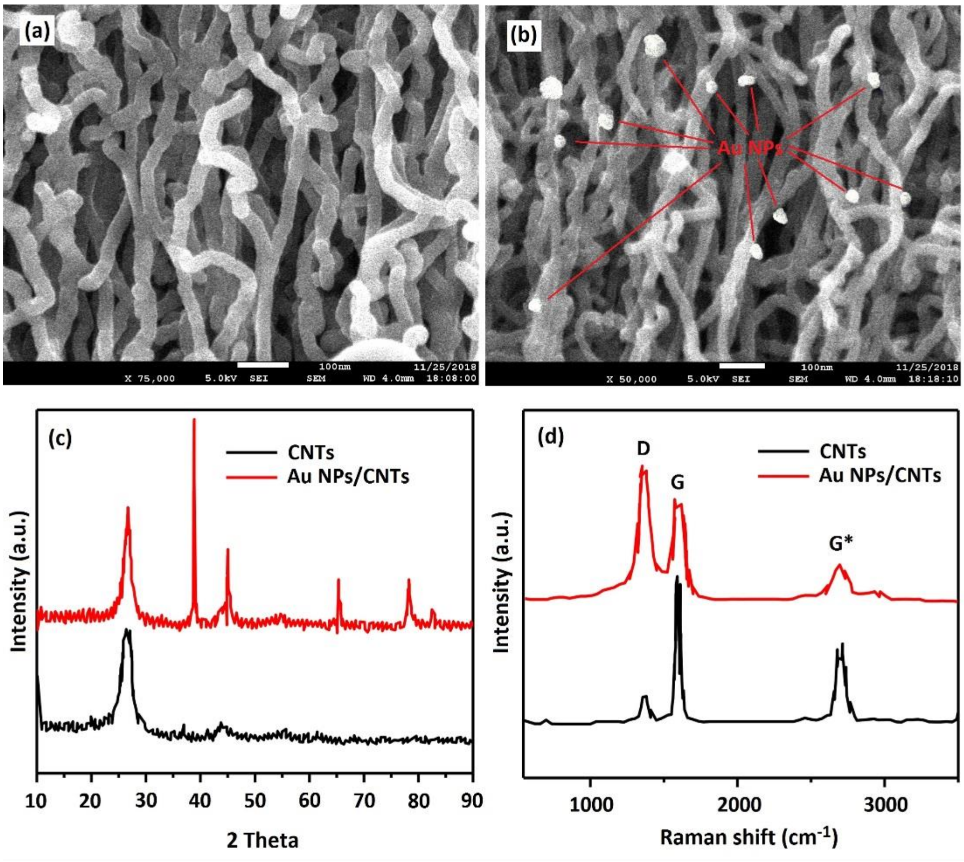

Figure 1. FESEM images of (a) CNTs (b) Au NPs/CNTs electrodes. (c) XRD pattern and (b) Raman spectra of synthesized CNTs and Au NPs/CNTs

Figure 1c shows the XRD pattern of CNTs and Au NPs/CNTs. XRD pattern in Figure 2 (a) gives a peak (002) at 25.9° corresponding to carbon nanotubes oriented along the c-axis and the obtained XRD spectrum is in good agreement with the standard JCPDF data reference (No. 58-1638) [20]. The XRD patterns of Au NPs/CNTs confirmed the presence of face-centered cubic (fcc) structured Au nanoparticles (JCPDS No 4-784) beyond the CNTs structure.

The Raman spectra of synthesized CNTs and Au NPs/CNTs are shown in Figure 1d. The spectral lines in the region of 1323 – 1334 cm^{-1}, 1570 – 1600 cm^{-1}, and 2630 – 2680 cm^{-1} named as D-line, G-line and G*-line, respectively [21]. The strong G-lines in the region of 1570 –1600 cm^{-1} in all captured spectra identify the presence of highly graphitized structures in the synthesized CNTs samples [22]. D-lines at 1330 cm^{-1} indicates the existence of the disordered carbon such as defective graphitic structure of CNTs [23, 24]. The relative intensity ratio of these bands (I_D/I_G) were estimated to be 0.45 and 1.1 for CNTs and Au NPs/CNTs, respectively. The higher I_D/I_G value indicates a greater disruption of the sp^2 – bonded carbon atoms to sp^3– hybridized carbon atoms suggesting that the species have been immobilized on the surface of the CNTs. This further suggests that the decoration of Au NPs took place on the surface of the CNTs

The electrochemical characteristic of CNTs and Au NPs/CNTs electrodes was studied using cyclic voltammetry. Figure 2 shows recorded cyclic voltammograms of CNTs and Au NPs/CNTs electrode in 0.1 M phosphate buffer solution pH 7.0 at a scan rate of 20 mV s^{-1} in the potential range of 0 to 1.0 V. As shown, no oxidation or reduction peak is observed for the electrodes in absent of phenol in the electrolyte. As shown in Figure 2, when 0.1 mM of phenol solution was injected into the electrochemical cell solution, recorded cyclic voltammograms showed sharp oxidation peaks at 0.57 V for each electrode. There is more current for the observed oxidation peak in Au NPs/CNTs electrode that refers to a higher sensitivity response to phenol determination [25, 26].

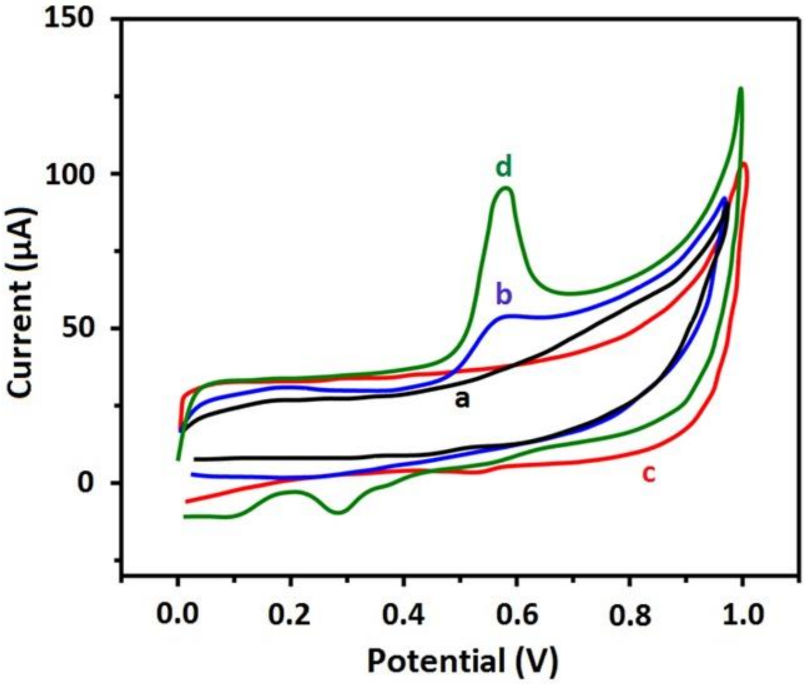

Figure 2. Recorded cyclic voltammograms in 0.1 M phosphate buffer solution pH 7.0 at a scan rate of 20 mV s^{-1} in absent of phenol solution for (a) CNTs and (c) Au NPs/CNTs electrodes, respectively. Recorded cyclic voltammograms after injection of 0.1 mM phenol solution for (b) CNTs and (d) Au NPs/CNTs electrodes, respectively.

In order to study the stability of CNTs and Au NPs/CNTs electrodes for oxidation behavior of phenol, the successive cycle voltammograms of 0.1 mM of phenol in 0.1 M phosphate buffer solution pH 7.0 at a scan rate of 20 mV s^{-1} were recorded. Figure 3 shows the 100th and 150th recorded successive cycle voltammograms. As shown in figure 3, the changes of phenol oxidation current in 0.57 V for CNTs and Au NPs/CNTs are more than 28% and less than 4%, respectively. In addition, Au NPs/CNTs electrode surface shows more stable response to the determination of phenol. Therefore, it can be considered that the electrodeposition or polymerization effect of phenol on Au NPs/CNTs surface was not performed. Because electrodeposition or polymerization of phenol on Au NPs surface leads to a formation of a thick film that can change significantly the oxidation peak current [27, 28]. Therefore, the oxidation current peak of phenol electrochemical activity on Au NPs/CNTs electrode surface is stable.

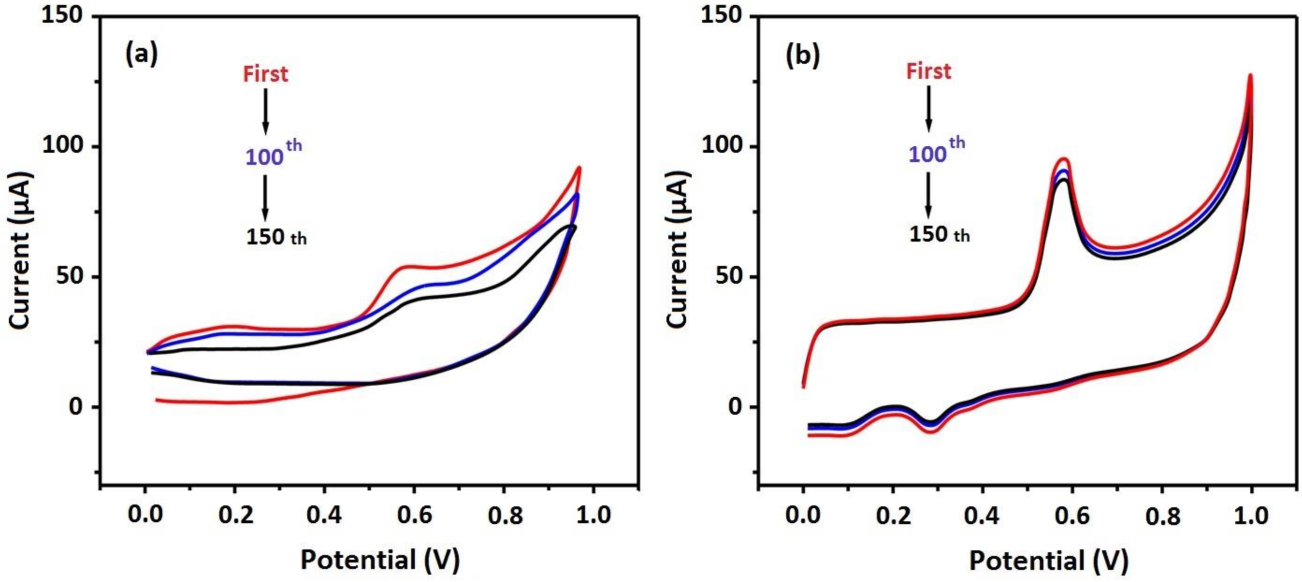

Figure 3. First, 100th and 150th Recorded cyclic voltammograms of (a) CNTs and (b) Au NPs/CNTs electrodes in 0.1 M phosphate buffer solution pH 7.0 at a scan rate of 20 mv s^{-1} after injection of 0.1 mM phenol solution.

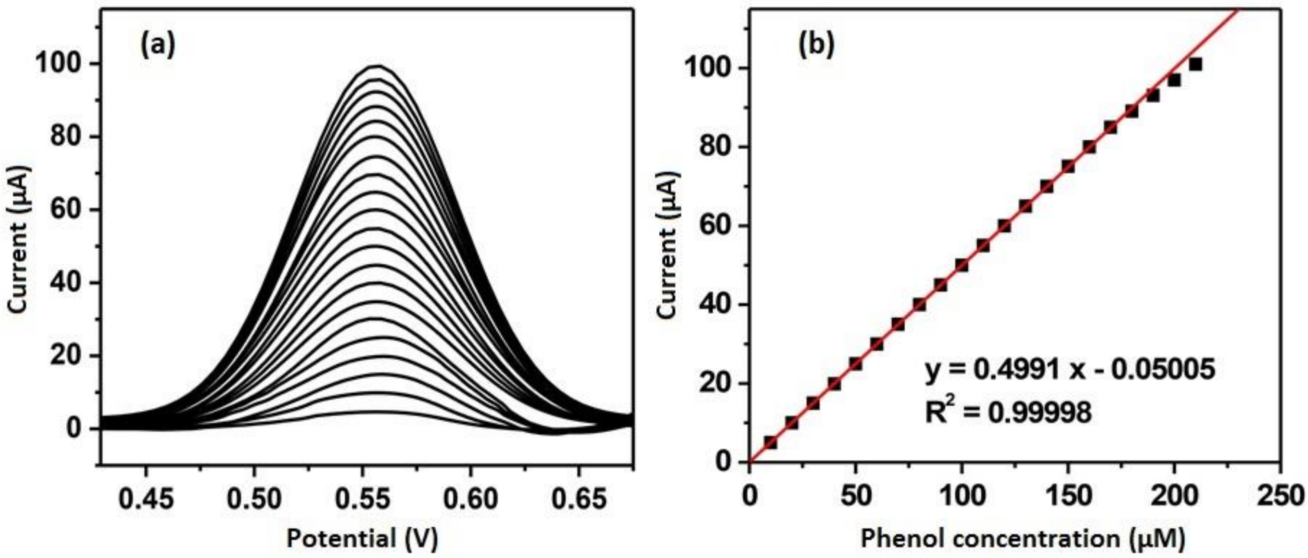

Figure 4. (a) The recorded differential pulse voltammograms of Au NPs/CNTs electrode in 0.1 M phosphate buffer solution pH 7.0 in successive additions of 10 µM phenol solution; (b) the plots of catalytic current vs. phenol concentration.

Differential pulse voltammetry as the electrochemical technique is sensitive approach to investigate the sensor properties such as detection limit, sensitivity, and selectivity. Figure 4a shows the recorded differential pulse voltammograms of Au NPs/CNTs electrode in 0.1 M phosphate buffer solution pH 7.0 in successive injections of 10 µM phenol solution. The limit of detection was calculated from the calibration curve as $3\sigma/a$ [29] where: σ is a standard deviation of the y-intercept and a is a slope of the calibration curve. As shown in figure 4b, the calibration curve for phenol measurement had a linear regression equation in the studied ranges (Y= 0.4991X - 0.05005) (the square of correlation coefficient were more than 0.98). Under these conditions a detection limit of 6 nM with a relative error of 4.6% and a standard deviation of 7.8% can be obtained. Furthermore, the sensitivity value was 0.499 µA/ µM.

A comparison in the performance of phenol electrochemical sensors with previous studies is shown in Table 1. The results illustrate that the Au NPs/CNTs sensor has low detection limit which are superior to the other reported phenol electrochemical sensors because of high porosity and especially high stability of gold nanostructure on CNTs and high porosity. It can be seen that DPV technique exhibited a relatively broader detection range and high sensitivity. Although the sensitivity was not as good as some analytical methods such as amperometry [30] due to rotating the electrode. Moreover, some researchers show a much larger determination range compared to the present study because they focused on the high concentration determination of phenol [31]. Moreover, comparison with other electrodes [30-37], the Au NPs/CNTs electrode is an excellent and alternative electrochemical sensor for the detection of phenolic compounds.

Table 1. Comparison in performance of phenol sensors.

Electrodes	**Technique**	**Electrolyte pH**	**detection limit (µM)**	**Linear range (µM)**	**Sensitivity (µA/µM)**	**Ref.**
Magnetic oxide/amino-functional graphene nanocomposite	Inear sweep voltammetry	9.78	0.4	156 -456	0.485	[30]
TiO2 nanoparticles/Mn(III) salen doped carbon paste electrode	Square wave voltammetry	7.0	0.01	0.03-400	0.101	[31]
Platinum nanoparticles modified graphite-like carbon nitride	Amperometry	3.0	0.667	2 - 20	1210.84	[32]
Tyrosinase-ZnO nanorods/Au	Amperometry	7.0	0.6	0.6–55	0.103	[33]
Poly(zincon) electrode	Voltammetry	7.0	9	21–292	0.0245	[34]
Nanocrystalline cellulose /CdS quantum dots nanocomposite	Differential pulse voltammetry	7.0	0.082	5–40	0.078	[35]
Reduced graphene oxide-zinc oxide composite modified glassy carbon electrode	Differential pulse voltammetry	7.2	1.94	2–15	1.79	[36]
Horseradish peroxidase/ reduced graphene oxide/ glassy carbon electrode	Differential pulse voltammetry	7.0	2.19	3–100	0.1669	[37]
Au NPs/CNTs electrode	Voltammetry	7.0	0.006	1–200	0.499	This work

In order to study the selectivity of Au NPs/CNTs electrode, the determination of phenol was investigated in the presence of benzene, nitrobenzene, glucose, gluconic acid, As^{3+}, Ni^{2+}, Al^{+3}, pd^{+2}, Mg$^{+2,}$ and SO$_3^{2-}$ analytes. Figure 5 illustrates the recorded differential pulse voltammograms of Au NPs/CNTs electrode in 0.1 M phosphate buffer solution pH 7.0. As shown, four successive injections of 10 µM phenol followed by successive injections of 10 µM of benzene, nitrobenzene, glucose, gluconic acid, and As^{3+} analytes, and then three successive injections of 10 µM phenol solution. Then, successive additions of 10 µM of Ni^{2+}, Al^{+3}, pd^{+2}, Mg$^{+2,}$ and SO$_3^{2-}$ ions were performed. Finally, two successive injections of 10 µM phenol were done. The results indicate that the modified electrode showed a clear response to all injections of phenol solution. The modified electrode did not reveal any significant response for the injections of other analytes. Furthermore, the modified Au NPs/CNTs electrode indicated the selective determination of phenol in the presence of the above analytes. Therefore, the above analytes do not have any interference with phenol determination on Au NPs/CNTs electrode surface.

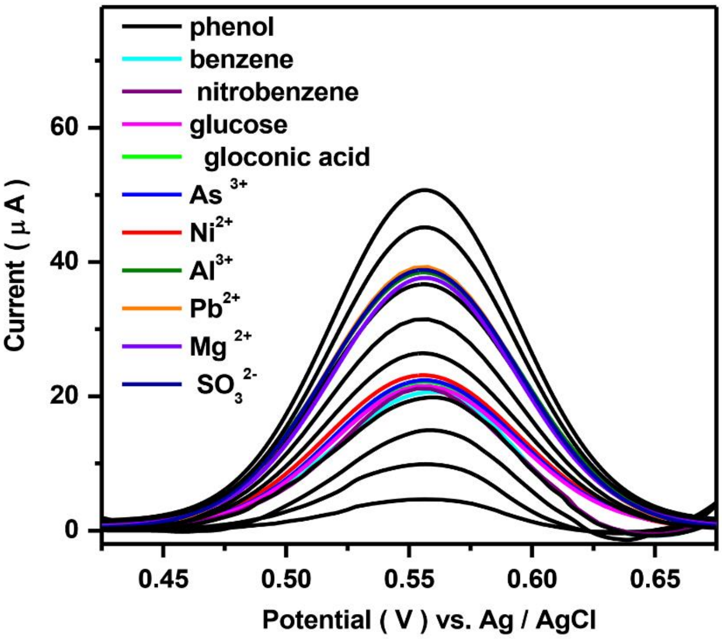

Figure 5. The recorded differential pulse voltammograms of Au NPs/CNTs electrode in 0.1 M phosphate buffer solution pH 7.0 at scan rate 10 mV s^{-1} for 10 µM successive additions of phenol and benzene, nitrobenzene, glucose, gluconic acid and As^{3+}, Ni^{2+}, Al^{+3}, pd^{+2}, Mg$^{+2,}$ and SO$_3^{2-}$ solutions.

In order to study the Au NPs/CNTs electrode for the determination of phenol in real samples, the concentration of phenol was measured in prepared sample from Xiangjiang River in Changsha, China. The real industrial wastewater was prepared using river water in 0.1 M phosphate electrolyte solution (pH 7.0). The successive injections of 0.01 µM phenol were added to the electrolyte. The standard injection of phenol was applied to determine the phenol in the sample. Figure 6a shows the recorded differential pulse voltammograms of the Au NPs/CNTs electrode in the sample for different successive injection of phenol. Figure 6b reveals the calibration graph as an equation of the current versus phenol concentration. As shown in figure 6b, the calibration curve for real phenol measurement had a linear regression equation (Y= 0.000549X - 0.009009) (R^2= 0.99933). Under these conditions a detection limit of 16 nM with a relative error of 2.3% and a standard deviation of 3.8% can be obtained. The sensitivity value in the real sample was 0.00054 µA/ nM. These results is very close to the report by Guan et al. [38]. It confirmed the ability of Au NPs/CNTs electrode to detect phenolic compounds in the real samples.

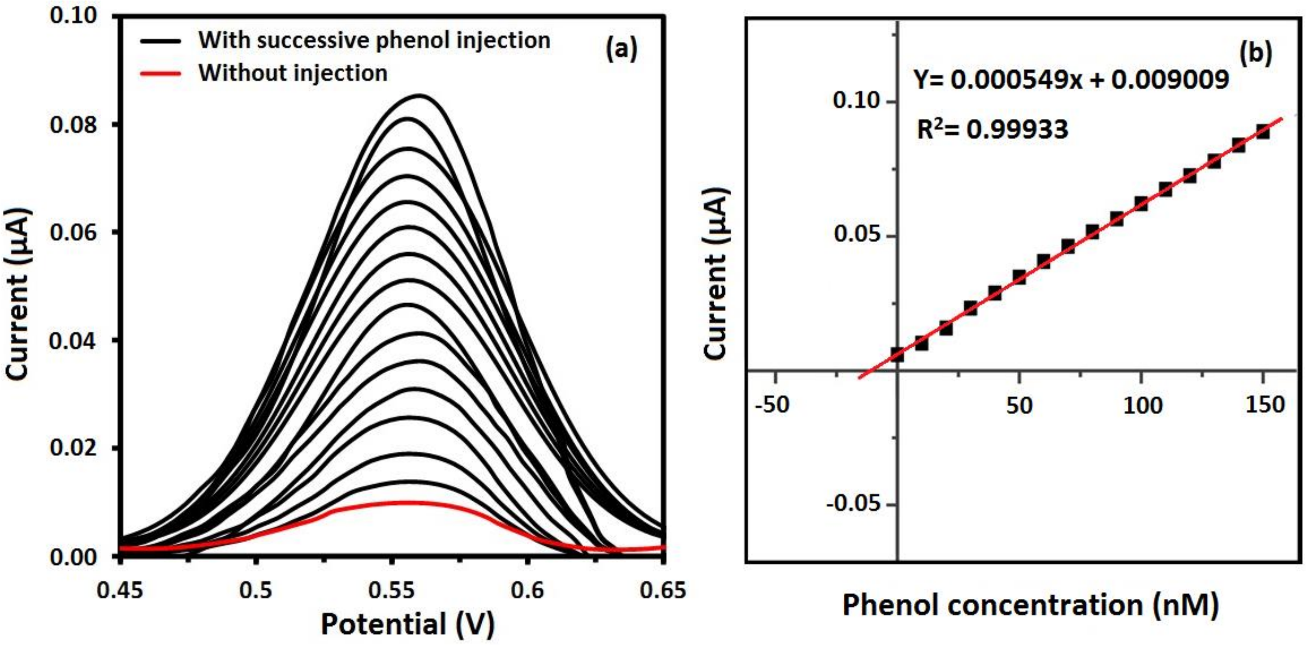

Figure 6. (a) The recorded differential pulse voltammograms of Au NPs/CNTs electrode in 0.1 M phosphate buffer solution pH 7.0 in the presence of different concentrations of phenol in a real sample at a scan rate of 20 mV s^{-1}; (b) Plot of calibration curve.

4. CONCLUSION

This study focused on the detection of phenolic compounds as pollutant in the wastewater of petroleum industry using Au NPs/CNTs electrode. The electrochemical deposition was applied for decoration of Au nanoparticles on synthesized CNTs. The structural properties of synthesized CNTs and Au NPs/CNTs were studied by SEM, XRD, and Raman analyses. Electrochemical properties and sensing properties of CNTs and Au NPs/CNTs electrode were investigated to detect the phenol by cycle voltammetry and differential pulse voltammetry techniques. Results of morphology and structure studies showed high density of Au NPs/CNTs with high aspect ratio and high effective surface area were synthesized on substrates. The CNTs were grown along the c-axis of crystallographic orientation and fcc structured Au nanoparticles deposited on the surface of CNTs structure. The result of cycle voltammetry study showed the stability behavior of Au NPs/CNTs electrode for the oxidation of phenol. Differential pulse voltammetry studied indicated Au NPs/CNTs electrode sensor was sensitive, selective and stable in the determination of phenolic compounds. The linear range, sensitivity, and detection limit of sensor were estimated 1-200 µM, 0.499 µA/ µM and 6 nM, respectively. Finally, the proposed method was successfully applied to phenol detection in real wastewater samples.

ACKNOWLEDGEMENT
This research was supported by the Fundamental Research Funds for the Undergraduate Universities of HeiLongJiang Province (2018-KYYWF-1182).

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