

Electrochemical Sensing of Phenol in Different Water Sources by a Titanium Oxide Nanotubes/Single-Wall Carbon Nanotubes Nanocomposite-Ionic Liquid Amplified Sensor

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Phenol and its derivatives are one of the main important pollutants of water sources. Highly sensitive electrochemical sensor modified with titanium oxide nanotubes/single-wall carbon nanotubes nanocomposite and room temperature ionic liquid (1-hexyl 3-methyl imidazolium hexafluorophosphate) (TiO₂/NT/SWCNTs/IL/CPE) was fabricated for the determination of phenol in well water, seawater and urban water samples. The TiO₂/NT/SWCNTs nanocomposite synthesized with thermal process and characterized with FESEM and TEM methods. Ration of mediators and pH value of solution were optimized as important factors on sensor ability for phenol determination and results confirmed best condition were obtained at pH= 6.0, 15% (v:v; 1-hexyl 3-methyl imidazolium hexafluorophosphate/paraffin oil) and 6% (w:w; TiO₂/NT/SWCNTs/graphite powder). In continuous, the recorded differential pulse voltammograms (DPV) confirmed that the electro-oxidation current of phenol improved about 2.9 times by TiO₂/NT/SWCNTs/IL/CPE compare to CPE and the phenol oxidation potential decrease about 105 mV. Then, the interference species on the determination of phenol evaluated and reported results showed high selectivity of TiO₂/NT/SWCNTs/IL/CPE for sensing phenol in water samples. DP voltammograms show wide linear dynamic ranges between 0.1 – 300 μM and a good limit of detection of about 0.05 μM for sensing phenol. Finally, TiO₂/NT/SWCNTs/IL/CPE is used to determine phenol in different water samples with acceptable recovery data.

Keywords: Phenol analysis, Titanium oxide nanotubes, Voltammetry, Single wall carbon nanotubes

1. INTRODUCTION

Phenol is an aromatic organic compound with high toxicology and application in different industries with the molecular formula C_6H_5OH and white crystalline solid. Phenol and its derivatives are synthesized from petroleum-derived feedstock's and essential for making plastics, polycarbonates and epoxide resins [1]. Due to a protein-degenerating effect, eye or skin contact with phenol or its vapors can cause serious health problems. If researchers use phenol and its combination for long time, our skin may cause dermatitis, or even second and third-degree burns. Due to its high toxicity as well as the widespread use of phenol in industry, its continuous measurement in water and wastewater samples has become of particular importance for controlling industrial and environmental conditions [2]. There are many methods for determination of phenol such as spectrophotometry [3], clean-up technique [4], HPLC [5, 6], pervaporation–flow injection analysis [7], and electrochemical sensors [8,9], solid-phase extraction method [10]. Among the above analytical methods, electrochemical methods were used in the recent years due to low cost, easy operation, rapid analysis, simple and portable strategies [11-25]. In the recent decade, researchers tried for improving of electrochemical sensors by modification of them by conductive mediators [26-30]. For this goal, different types of mediators such as DNA, nanomaterials, ionic liquids, polymers checked and ionic liquids and nanomaterials showed more activity in this regards [50-60].

Nanomaterials have gained a special place in the sciences by creating new and incredible properties of materials [61-76]. One of the unique features of nanomaterials, especially carbon and metal nanomaterials and their composites, is their high electrical conductivity [77-80]. This issue has caused this category of materials to be considered by many researchers in the field of sensors. In this project, research team decide to fabricate new and sensitive electrochemical modified sensor with $TiO_2/NT/SWCNTs$ nanocomposite and 1-hexyl 3-methyl imidazolium hexafluorophosphate for determination of phenol in some real waters such as well water, urban water and sea water. The $TiO_2/NT/SWCNTs/IL/CPE$ showed catalytic activity on oxidation signal of phenol and used for sensing of these pollutants in the nanomolar concentration range.

2. EXPERIMENTAL SECTION

2.1. Reagents and Apparatus

The entire reagent used in this research includes phenol, 1-hexyl 3-methyl imidazolium hexafluorophosphate, sodium hydroxide, phosphoric acid, nitric acid paraffin, graphite and ethanol were purchased from Merck Company. An Ivium-Vertex that connected to the computer with IviumSoft software included three electrochemical cells included $TiO_2/NT/SWCNTs/IL/CPE$ (as working electrode), $Ag/AgCl/KCl_{sat}$ (as references electrode) and Pt wire (as a counter electrode) was used for electrochemical investigation.

2.2. Preparation of $\text{TiO}_2/\text{NT}/\text{SWCNTs}/\text{IL}/\text{CPE}$

For preparation of $\text{TiO}_2/\text{NT}/\text{SWCNTs}/\text{IL}/\text{CPE}$, 0.94 g graphite powder + 0.06 g $\text{TiO}_2/\text{NT}/\text{SWCNTs}$ was mixed together by hand mixing. 10 mL pure ethanol was added to the mortar to ensure complete mixing of the material during the grinding operation. After evaporation of ethanol, paraffin oil and 1-hexyl 3-methyl imidazolium hexafluorophosphate with ratio 85%:15% (v:v) added to powders as binders and sample hand mixed for 30 min. Resulting paste filled it in to the glass tube in the presence copper wire.

2.3. Preparation of stock solution

To prepare a 0.01 M phenol solution as a stock solution for electrochemical investigation, 0.094 g of phenol was dissolved into 100 mL distilled water using volumetric flask and used during this project.

2.4. Real samples preparation

Some of the different samples of city water, seawater and well water are taken and centrifuged and then filtered. The obtained sample is used as a test sample to determine phenol at surface of $\text{TiO}_2/\text{NT}/\text{SWCNTs}$.

3. RESULTS AND DISCUSSION

3.1. Characterization of $\text{TiO}_2/\text{NT}/\text{CNTs}$ nanocomposite

FESEM and TEM methods were suggested for characterization of $\text{TiO}_2/\text{NT}/\text{SWCNTs}$ nanocomposite and relative images are presented in Figure 1 A&B. In FESEM and TEM images, the presence of SWCNTs mixed TiO_2 can be observed.

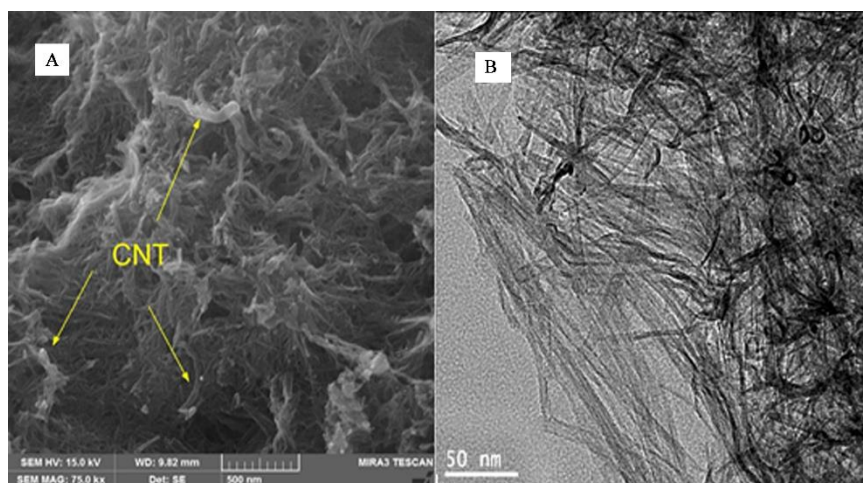


Figure 1. A. FESEM image of $\text{TiO}_2/\text{NT}/\text{SWCNTs}$ and B) TEM image of $\text{TiO}_2/\text{NT}/\text{SWCNTs}$

3.2. Voltammetric investigation

At first step, the electro-oxidation behavior of phenol investigated by differential pulse voltammetric method at a different pH of phosphate buffer solution. According to the previous research, we guessed that the phenol oxidation would be pH dependent on one proton's electro-oxidation and one electron. In order to this, the voltammetric response of 150 μM phenol at a surface of $\text{TiO}_2/\text{NT}/\text{SWCNTs}/\text{IL}/\text{CPE}$ was obtained in buffer solutions with different pH value from 4.0 – 8.0. Result confirm our thought, the peak potential of the redox couple was pH dependent with a slope of -0.051 V/pH unit at 25°C which was equal to the Nernstian value for one-electron and one-proton electrochemical reaction. The results are in good agreement with the reports of other researchers [81]. Results show in (Figure 2) and pH = 6.0 was selected as an optimized condition during the work.

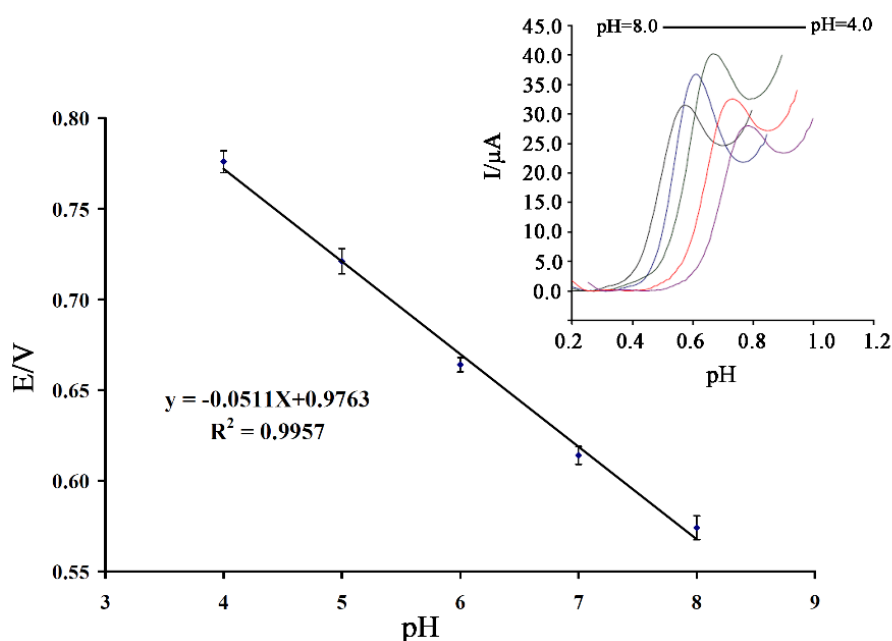


Figure 2. Plot of E Vs pH of 150 μM phenol at surface of $\text{TiO}_2/\text{NT}/\text{SWCNTs}/\text{IL}/\text{CPE}$, inset: differential pulse voltammograms of 150 μM phenol at pH ranges 4.0 – 8.0.

3.3. Catalytic effect

Catalytic effect is another important parameter of a new sensor that was investigated in this step. Differential pulse voltammograms of 150 μM phenol at the surface of different electrodes at pH 6.0 was recorded. Results showed in Figure 3 (curves a-d) and recording signal at unmodified electrode (CPE) showed oxidation peak potential $\sim 780 \text{ mV}$ with the oxidation peak current of $13.8 \mu\text{A}$ (Figure 3, curve a). The oxidation current of phenol was increased to $27.6 \mu\text{A}$, $32.1 \mu\text{A}$ and $40.2 \mu\text{A}$ and oxidation potential of phenol was decrease to 742 mV , 709 mV and 675 mV at surface of $\text{TiO}_2/\text{NT}/\text{SWCNTs}/\text{CPE}$ (curve b), IL/CPE (curve c) and $\text{TiO}_2/\text{NT}/\text{SWCNTs}/\text{IL}/\text{CPE}$ (curve d), respectively. By comparing the reported curves, a better oxidation signal of phenol can be observed at the surface of $\text{TiO}_2/\text{NT}/\text{SWCNTs}/\text{IL}/\text{CPE}$, improving about 2.9 times in current. These results confirm

TiO₂/NT/SWCNTs' high conductivity and 1-hexyl 3-methyl imidazolium hexafluorophosphate as two conductive mediators [82, 83].

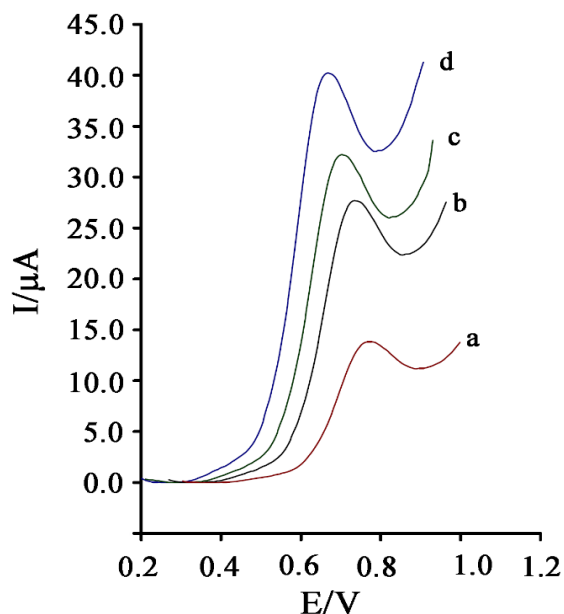


Figure 3. Differential pulse voltammograms of 150 μM phenol at surface of a) CPE, b) TiO₂/NT/SWCNTs/CPE, c) IL/CPE and d) TiO₂/NT/SWCNTs/IL/CPE.

3.4. Scan rate

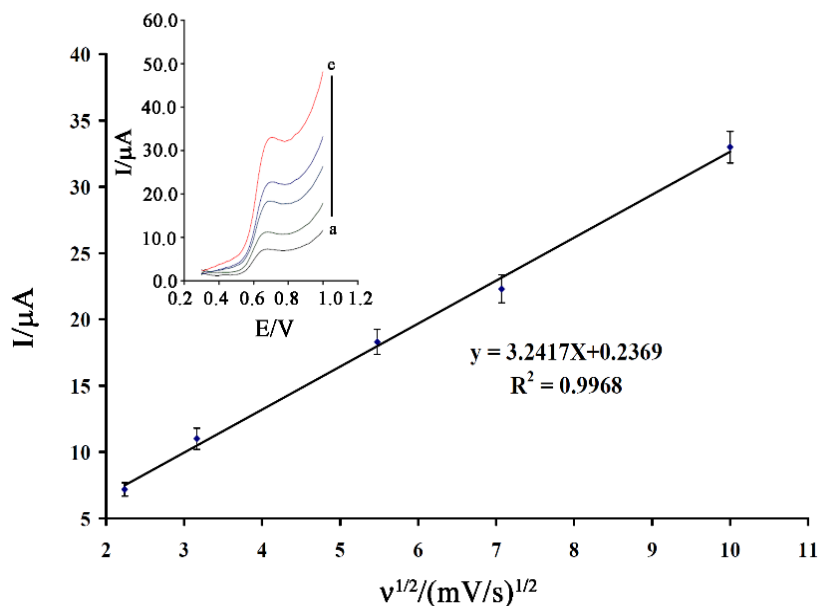


Figure 4. Plot of I_p vs. $v^{1/2}$ at the surface of TiO₂/NT/SWCNTs/IL/CPE in the presence of phenol (pH=6.0). Inset linear sweep voltammograms of TiO₂/NT/SWCNTs/IL/CPE in the presence of phenol at different scan rate ranges: a) 5.0, b) 10.0, c) 30.0, d) 50.0 and e) 100.0 mV/s

The scan rate effect was investigated in the range of 5–100 mV s^{-1} at the surface of $\text{TiO}_2/\text{NT}/\text{SWCNTs}/\text{IL}/\text{CPE}$ (Figure 4). There is a relationship between the peak current (i_p) and $v^{1/2}$ regarding the equation that shows the diffusion-controlled [83-86] process for the electro-oxidation of phenol on the surface of $\text{TiO}_2/\text{NT}/\text{SWCNTs}/\text{IL}/\text{CPE}$ in this work.

3.5. LOD and LDR investigation

Differential pulse voltammetric (DPV) strategy was used to determine the linear dynamic ranges of phenol using $\text{TiO}_2/\text{NT}/\text{SWCNTs}/\text{IL}/\text{CPE}$ as sensor (Figure 5). Results clearly showed a linear dynamic range between 0.1 - 300.0 μM with equation $I = 0.2477 C_{\text{Phenol}} + 4.3221$ ($R^2 = 0.9935$) and limit of detection ($Y=3s_b/m$ [87]) 0.05 μM by DPV method.

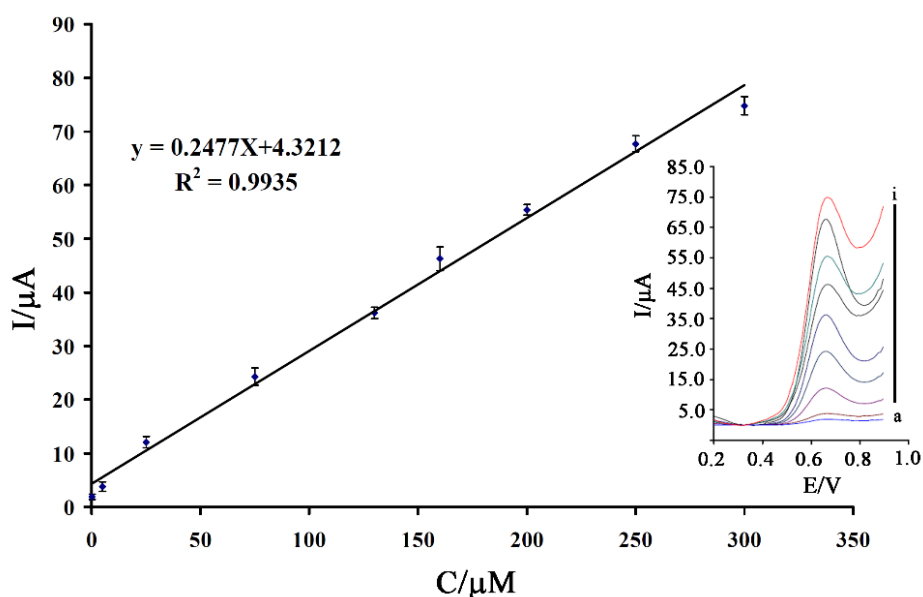


Figure 5. Plot of oxidation current of phenol vs. its concentration in the range of 0.1 - 300.0 μM . Differential voltammograms of phenol in the concentration range 0.1 - 300.0 μM at $\text{TiO}_2/\text{NT}/\text{SWCNTs}/\text{IL}/\text{CPE}$.

3.6. Interference Study

For evaluation the selectivity of $\text{TiO}_2/\text{NT}/\text{SWCNTs}/\text{IL}/\text{CPE}$ for determination of phenol, the interference of different foreign species that is usual in water samples was investigated in the presence of 15.0 μM phenol. The tolerance limit was evaluated as the maximum concentration of foreign substances with an approximate relative error of $\pm 5\%$. The results are shown in Table 1.

3.7. Real sample analysis

In order to determine the sensitivity of the modified sensor and its efficiency, samples of water such as well water, seawater and urban water were collected and use another method, standard addition, to confirm the applicability and selectivity and sensitivity of the proposed sensor. The results of the statistical calculation shown in Table 2.

Table 1. Interference study of 15.0 μM of phenol

Tolerance limits ($W_{\text{Substance}}/W_{\text{phenol}}$)	Species
300	Sulfide, thiosulfate, sulfite
250	4- Chlorophenol, 4- Nitrophenol, Hydrazine
500	Hydroxylamin
1000	Na^+ , Li^+

Table 2. Phenol analysis in water samples (n=3)

Sample	Added Phenol (μM)	Expected Phenol (μM)	Found phenol Phenol (μM)	Recovery (%)
Well water	---	---	<LOD	---
	10.00	10.00	10.45 ± 0.75	104.5
Sea water	---	---	<LOD	---
	20.00	20.00	20.45 ± 1.12	104.5
Urban water	---	---	<LOD	---
	5.00	5.00	4.89 ± 0.45	97.8

4. CONCLUSION

Combination of $\text{TiO}_2/\text{NT}/\text{CNTs}$ and 1-hexyl 3-methyl imidazolium hexafluorophosphate as a room temperature ionic liquid for sensor modification to determine the phenol in water samples is new and high sensitive electrode. Synthesized nanocomposite tested with FESEM, TEM images and XRD pattern. The pH optimized and electrochemical parameters are evaluated, too. The limit of detection of 0.05 μM and linear dynamic ranges between 0.1 – 300 μM showed the sensitivity of the proposed electrode for sensing phenol. It has good application in real samples and the statistic calculator indicates the reliability and applicability of the proposed sensor. Compared to other analytical methods for sensing phenol, the $\text{TiO}_2/\text{NT}/\text{SWCNTs}/\text{IL}/\text{CPE}$ showed more sensitivity, easy operation, and low cost.

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