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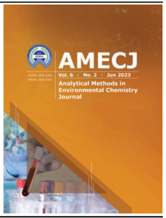

Modified carbon paste electrode based on nanotechnology for determining phenol in the liquid solutions by cyclic voltammetry and comparing to high-performance liquid Chromatography

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ABSTRACT

In this paper, phenol was determined in a liquid solution based on fabricating a phenol-selective electrode by cyclic voltammetry (CV). The carbon paste electrode was modified with nickel oxide nanoparticles (NiO) which were doped with nitrogen carbon quantum dots (NCQD) as the NiO-NCQD nanocomposite. The modified carbon paste electrode was manufactured in a laboratory and the effect of pH was studied. In the optimized condition, the best results were created at pH 7.0 and 4.0 using KH_2PO_4 buffer solution. By voltammetry, the voltage was optimized, and the best value for the voltages was obtained at 0.04166V and 0.05991V for pH 4 and 7, respectively. The scan rate (SR) was studied and the best SR was achieved at 100 mv s^{-1} for both pH. Due to the results, a wide linear dynamic range between 10 to 1000 µM was obtained. Also, the standard phenol solution was analyzed by high-performance liquid chromatography (HPLC). The retention time (RT), the wavelength maximum (λ max: nm), and the peak area equation of HPLC were achieved at 2.982 min, 270 nm, and ($Area=40420C_{Phenol}+43.557$), respectively by the concentration range of 0.1-5.0 mg L^{-1}. The modified carbon paste electrode with NiO-NCQD was used for determining phenol by cyclic voltammetry and compared with the HPLC technique. The results showed that there was no significant difference between the two methods.

1. Introduction

Phenol is an aromatic organic compound [1-2], it has the formula C_6H_6O [3], phenolic compounds are widely found in water, soil, and air. These compounds are not biodegradable [4], which are dangerous compounds, it is found everywhere in the environment, including in aerosols due to combustion emissions [5]. It is very toxic [6-8], phenol and its derivatives are pollutants [9], when a person is exposed to phenol for long periods, it enters his body through inhalation, ingestion, or through direct contact with the skin, and causes severe damage such as: including damage to the liver, kidneys, urinary and reproductive tracts, lungs, shortness of breath, neurological problems, severe abdominal pain, irritation of the digestive system, nausea, vomiting, diarrhea, and sweating, Coma and death. The lethal dose of phenol is ingestion of 1 g [10 -14]. Phenol disrupts endocrine function and, in addition, causes cancer [15], the maximum permissible level of phenol according to the world health organization (WHO) that its concentration does not exceed 0.1 µg L^{-1} in drinking water [16]. Phenol oxidizes to give an electron and a proton,

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and turns into hydroquinone and catechol, passing through radically [17]. Phenol and total phenol can be analytically spectro-photo-metrically in the visible (VIS), phenol and total phenol can be estimated spectro-photo-metrically in the visible range (VIS) based on the Folin-Denis detector or the Folin-Ciocalteu reagent (FCR) affected by phenolic hydroxyl and resulting in a blue compound that absorbs at different wavelengths between 620 nm and 740 nm, and maximum absorption reach to 765 nm. The reaction takes place in an alkaline medium by the influence of a saturated solution of sodium carbonate (Na_2CO_3). Folin-Ciocalteu reagent is affected by phenolic hydroxyl and results in a blue compound that absorbs at a wavelength ranging between (620-740) nm, and the maximum wavelength at which it absorbs may reach 765nm, the reaction occurs in an alkaline medium by being affected by a saturated solution of sodium carbonate ($Na_2 CO_3$) [18], phenol can also be analyzed in the ultraviolet (UV) at 270 nm [19], Phenol can also be analyzed in (HPLC) [20-24]. Carbon paste electrodes (CPE) are important for being chemically inert, electrode surface renewability, low ohmic resistance, easy to fabricate, environmentally friendly, and low cost. However, its stability, kinetics, and selectivity are weak. To solve this problem, surface modification (MCPE) is resorted to by modifiers [25], the modified carbon paste can be modified by adding several metallic or organic substances or a mixture of them. The oxides of metallic nanomaterials can also be used with carbon quantum dots by mixing them together and then adding them in an extended proportion to the modified carbon paste. Quantum carbon dots, nitrogen carbon quantum dots, and graphene quantum dots (GQDs) are used in several fields, such as the manufacture of solar cells [26-27], the environmental and analytical fields as determining ion Cu(II) and monitoring for bisphenol [28]. Some adsorbents were used to remove pollutants from environmental matrixes such as ethylbenzene [29], Pb(II) and Cd(II) ions [30], BTEX (Benzene, Ethylbenzene, Toluene, Xylenes), etc [31], they are also used in perchloroethylene vapor adsorption [32]. Another technology based on nanoadsorbents and ionic liquids was used for the removal/extraction of organic materials in different matrixes [33-37]. Cyclic voltammetry analysis is one of the important analytical methods through which the quantitative and qualitative analysis of the studied material can be mode, in addition to the possibility of studying the behavior of the studied material, for example, diffusion coefficient (D), charge transfer coefficient (α.nα), the mass transport (m_{trans}), constant K^0, Gibbs free energy (ΔG), Also, the highest occupied molecular orbital (HOMO), lowest unoccupied molecular orbital (LUMO), and others [17]. The research aims to fabricate a selective electrode for phenol based on carbon paste modified by (CV) method, which is a low-cost and easy-to-apply method in order to supplement the analytical methods in general and electrochemical in particular with a new method for determining phenol as one of the polluting and toxic organic compounds to the environment.

In this research, the electrochemical techniques based on newly modified carbon paste electrodes with nickel oxide nanoparticles (NiO-NCQD) were used for determining phenol in liquid solutions by cyclic voltammetry (CV) and comparing it to the HPLC method.

2. Experimental

2.1. Instrument

Voltammetry system for trace analysis and education. Complete accessories with VA Computrace software and all electrodes for a complete measurement system: Multi-Mode Electrode pro (MME pro), Ag/AgCl reference electrode, and Pt auxiliary electrode. In this study, a modern voltammetric was connected to a PC based on a USB port (Metrohm 797; volt-amperometric analyzer with analyzer cell). SHIMADZU SPP-M20A high-performance liquid chromatography HPLC device connected to a diode array detector with chromatographic column (C_{18}), with dimensions of 25cm×0.46cm×5µm. Sartorius pH meter type PB-11 was used from Data Weighing System Company (pH meter and mV meter; DWS Inc., USA).

2.2. Reagents

All chemicals used are characterized by high purity, namely: phenol (C_6H_6O) manufacturer ACROS, molecular weight 94.11 g mol^{-1}, specific density d=1.070 g cm^{-3}, high purity 99%, Potassium hexacyanoferrate(II) trihydrate (K_4 [$Fe(CN)_6$]. $3H_2O$) purchased from Sigma, Germany (CAS No.: 14459-95-1). Boric acid (H_3BO_3), phosphorous acid H_3PO_4, and sodium hydroxide (NaOH) were prepared by HiMedia, India (CAS No.: 1310-73-2). Monosodium di-hydrogen phosphate (NaH_2 PO_4) prepared from the LOBAL-Chemie Company, India (CAS No.: 7558-80-7). Also, mono-potassium di-hydrogen Phosphate (KH_2PO_4) was prepared from Sigma, Germany (CAS No.: 7778-77-0). Acetic Acid (CH_3COOH) Produced by Sigma, Germany (CAS No:64-19-7). Phosphorous Acid (H_3PO_4, 85%, 1.71g cm^{-3}) was purchased from the chemicals BDH, North America, (CAS No:7664-38-2). Glassware of various sizes, boiled and cooled double distilled water, glass tube, and copper wires was used.

2.3. General procedure

2.3.1.Fabrication of selective electrode NiO-NCQD/MCPE

The selective electrode is made (in the laboratory), it consists of a glass tube that is open at both ends and contains at its lower end modified carbon paste, At the upper end, it is connected to the device. A copper wire conducting electric current is connected between the modified carbon paste and the device. Then the electrode is connected to the volt-amperometric cell VA which consists of a working electrode (WE) and a comparison electrode and it is usually an Ag/AgCl electrode where its potential is 0.222v at 25°C and an auxiliary electrode (AE) (Fig. 1).

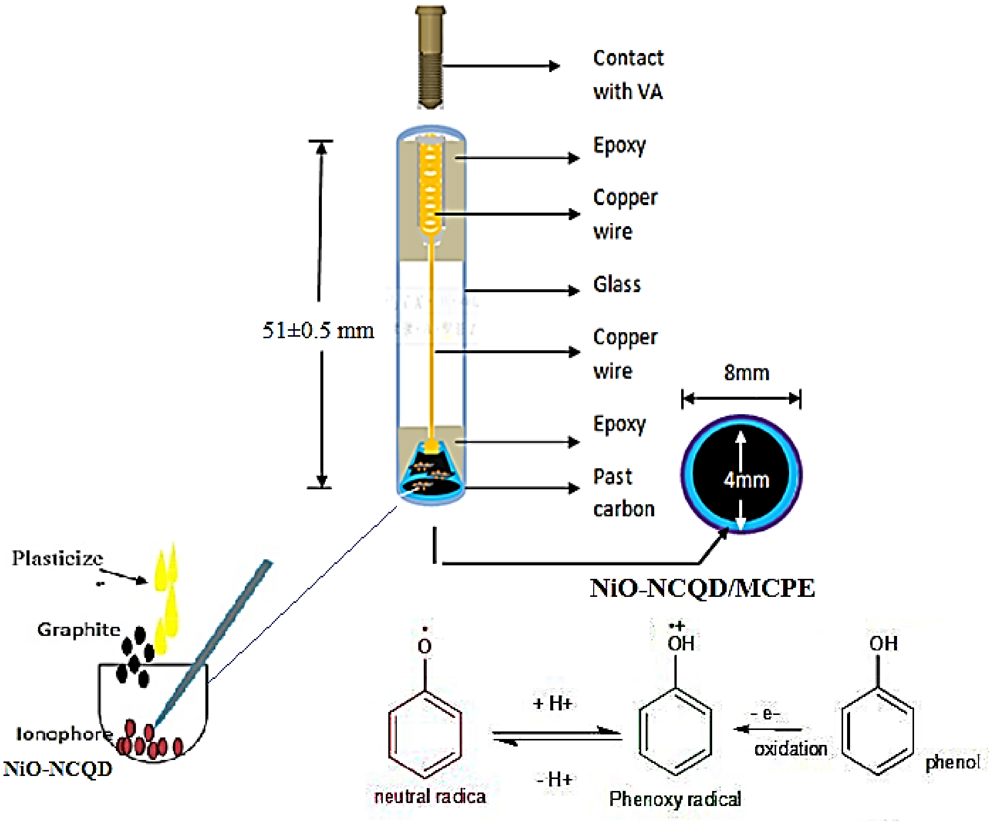


Fig. 1*:* Scheme of modified carbon paste components and electrode dimensions for phenol oxidation

2.3.2. The solution used for electrode testing

Test solution of 5m M of K4 [Fe(CN)$_6$].3H$_2$ O and 0.1 M from KCl.

2.3.3. Preparation of stock solution

To prepare a 0.1036 M of phenol solution, take 0.974 g of phenol then dissolved it into 100 mL of distilled water using a volumetric flask.

2.3.4. Preparation Modified carbon paste

Modified carbon paste using NiO-NCQD nanocomposite (3.0, 5.0, 7.0, 9.0, 12.0) %, graphite (48.5, 47.5, 46.5, 45.5, 44.0) % and paraffin oil (48.5, 47.5, 46.5, 45.5, 44.0) %, for a total weight of the modified carbon paste 0.5 g. The components are mixed in specific proportions and then packed in the electrode body made of glass, which symbolizes the factory electrode (NiO-NCQD/MCPE).

2.3.5. standard solution for HPLC

An amount of 0.0101 g of Phenol is taken and transferred to a 100 mL volumetric flask in double distilled water, its concentration is 101 mg L^{-1}. The solution is left to cool down and then the standard series is prepared (20.0, 5.0, 2.0, 1.0, 0.5, 0.1) mg.L^{-1}, filtered by a 0.45µm micro-membrane filter and then measured using high-performance liquid chromatography (HPLC). At a temperature of 40°C, the mobile phase is a mixture of (acetonitrile: water) in a volumetric ratio (80:20) v/v with a mobile phase flow of 1 mL per 1 min, and a chromatographic column of C$_{18}$ was used for HPLC.

2.3.6. Preparation of monosodium phosphate buffer solution

A phosphate buffer ($NaH_2 PO_4$) from a concentration of 0.1 M and a solution of NaOH (0.1M) was prepared by mixing different volumes of both of them to obtain a pH (4-8).

2.3.7. Preparation of mono-potassiumphosphate buffer solution

The buffer of $KH_2 PO_4$ at a concentration of 0.1M and a solution of potassium hydroxide (KOH, 0.1 M) was prepared by mixing different volumes of both of them to obtain a pH (4 and 7).

2.3.8. Preparation Britton –Robinson Buffer solution

Britton-Robinson buffer solution (BRB) containing $H_3 PO_4$ with a concentration of 0.04 M, acetic acid (CH_3COOH) at a concentration of 0.04 M, and boron acid ($H_3 BO_3$) with a concentration of 0.04M. Then, it was modified with a solution of NaOH with a concentration of 0.2 M to get the required pH value of 4.

2.3.9. Preparation of acetic buffer solution

The acetic acid at a concentration of 0.1 M was modified with a solution of NaOH with a concentration of 0.2 M to get the required pH value of 4.

3. Results and Discussion

3.1. Verification of the manufactured electrode

The manufactured electrode was tested using a solution test as in Figure 2.

3.2. The effect of the effective ratio on the electrode

The effect of the effective ratio on the electrode is studied with different proportions of nanoparticles from nickel oxide nanoparticles (NiO) doped with nitrogen carbon quantum Dots (NCQD), as was shown in Figure 3.

From the previous drawing curve, it was found that the best 12% of nickel oxide nanoparticles loaded with NiO-NCQD nanocomposite with graphite powder 44%, plasticizer, and paraffin oil 44%. The total weight of the modified carbon paste is 0.5 g based on 0.06 g of NiO-NCQD, 0.22 g of graphite, and 0.22 g of paraffin oil.

3.3. The effect of pH

The effect of pH is studied using a buffer solution of NaH_2PO_4 within a pH range from 3 to 8 by cyclic voltammetry based on modified carbon paste (NiO-NCQD) nanocomposite (Fig. 4).

It was found through the previous curve that for the phenol oxidation curves, a potential oxidation peak appears at 0.43V for a pH = 7,

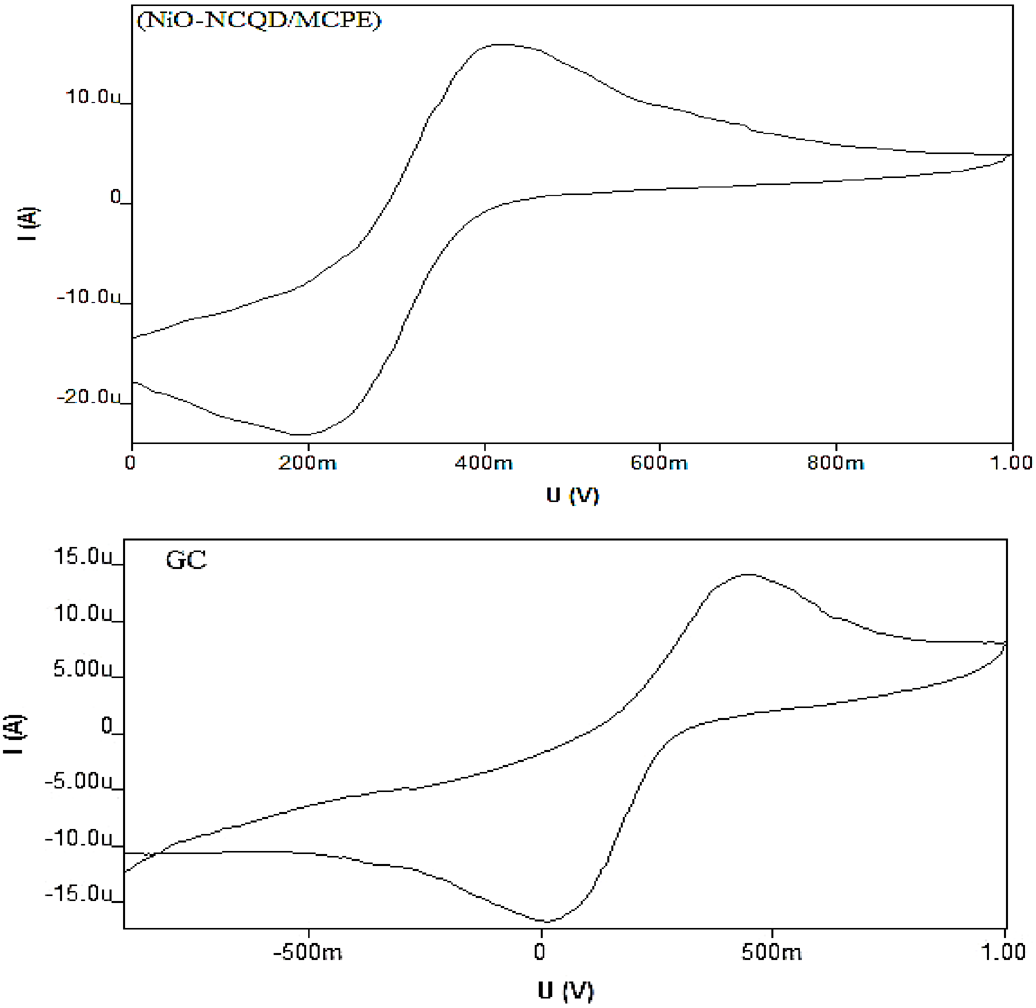


Fig 2: Oxidation of ferric ions in 5mM and 0.1M using Electrode glass carbon (GC) Electrode manufactured (NiO-NCQD/MCPE)

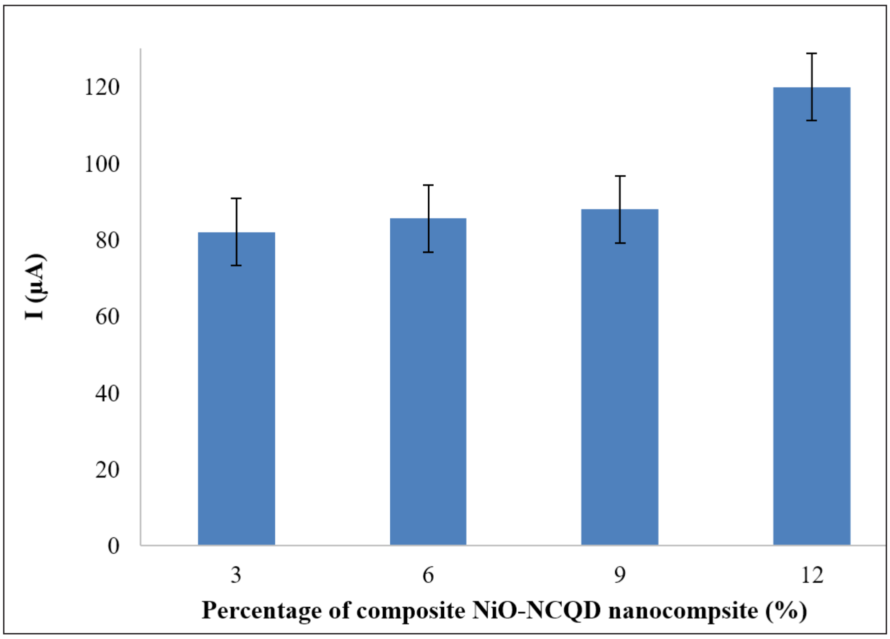


Fig. 3. The effect of the effective ratio (NiO-NCQD nanocompsite%) on I(µA) using the proposed electrode (NiO-NCQD/MCPE)

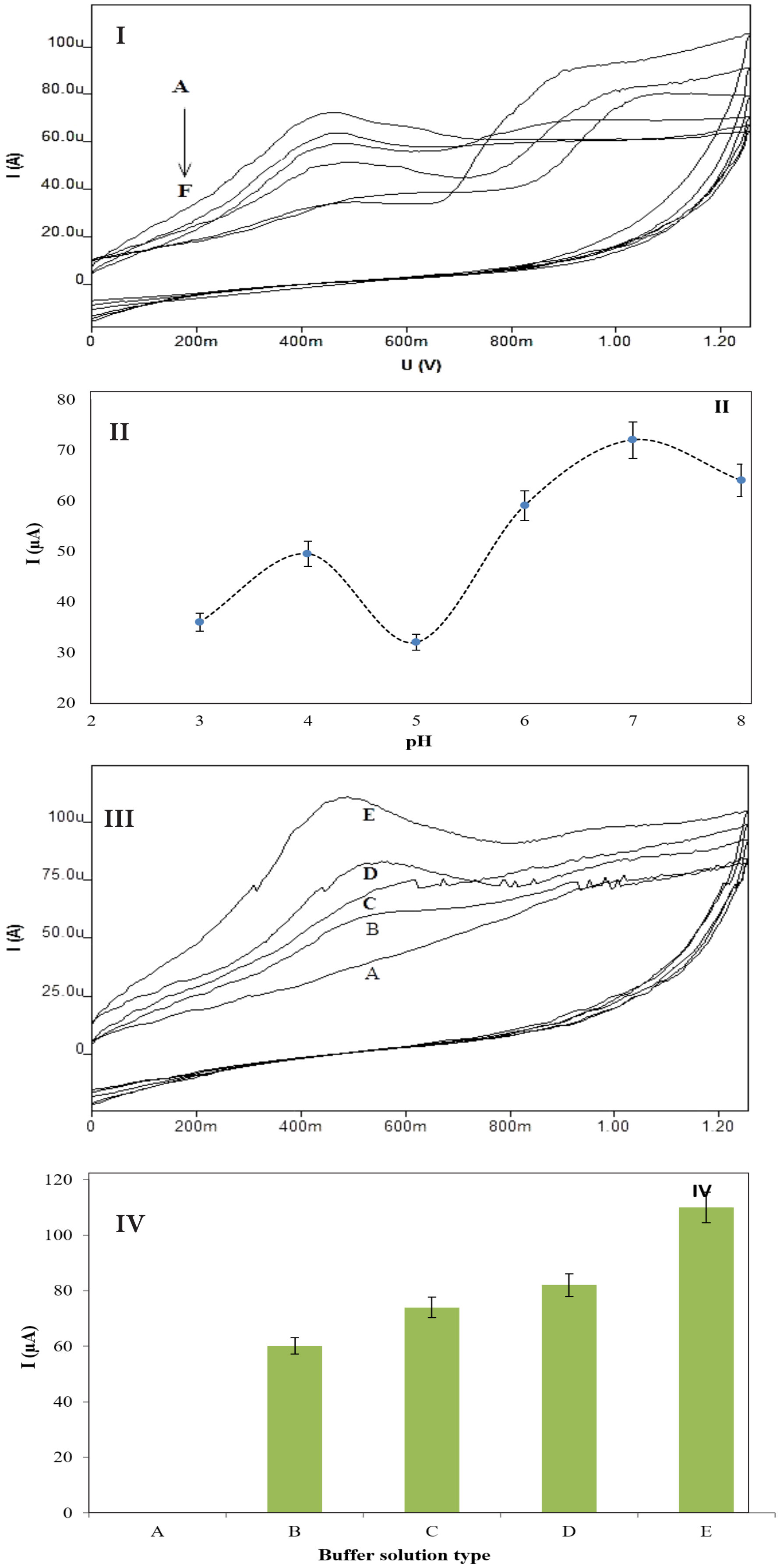


Fig. 4. Current oxidation for 1 mM phenol on the proposed electrode (NiO-NCQD/MCPE),

(I) Effect of $NaH_2 PO_4$ buffer solution from pH (3 to 8) pH values A) 7; B) 8; C) 6; D) 4; E)5; F)3,

(II) Effect pH vs I (µA),

(III and IV) Effect of the type of solution on the peak current 1 mM phenol A: Acetic buffer, B: $NaH_2 PO_4$ with citric acid buffer C: BRB, D: $NaH_2 PO_4$ buffer, E: $KH_2 PO_4$ buffer using the proposed electrode (NiO-NCQD/MCPE).

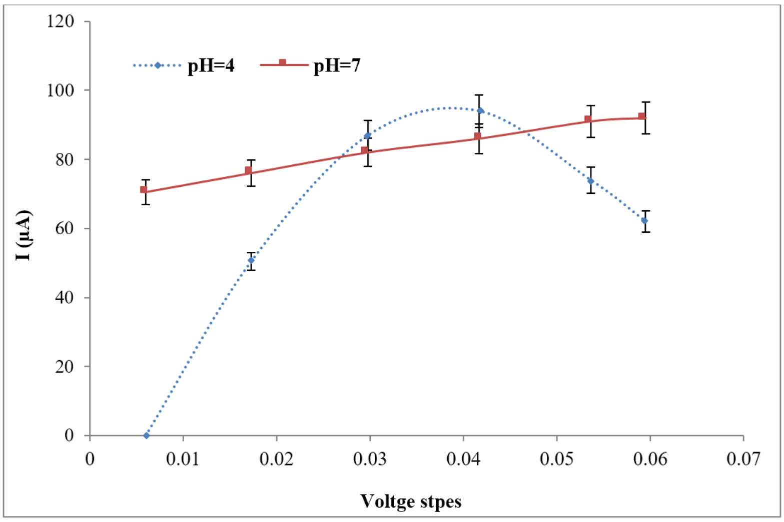


Fig 5. Effect of step voltage for 1 mM phenol using buffer at pH= 4 and pH= 7 using the proposed electrode (NiO-NCQD/MCPE)

while two oxidation peaks appear at 0.43V and 1.04V for a pH = 4, the peak current in terms of pH are studied in Figure 4. From the previous drawing curve, through the values of i_p and *U(V),* it was noted that at its peaks i_p=49.5µA, and 72µA at pH =4,7 respectively, so these two values are adopted. In the case (CV) method for phenol, it undergoes an oxidation process only without reduction, so the system is irreversible. Phenol oxidation is one-electron oxidation, the effect of buffer solutions was studied, as shown in Figure 4. From the previous drawing curve, the mono-potassium phosphate buffer gives the highest peak current, so the best buffer is the $KH_2 PO_4$ buffer.

3.4. step voltage effect

The step voltage was studied at the pH (4,7) within voltage steps (0.00595, 0.017253, 0.029755, 0.04166, 0.053599, 0.0595) voltage steps which was shown in Figure 5.

From the previous drawing curve, the best value for the step voltage was 0.04166V and 0.05991V for both (pH =4,7) respectively, noticed two peaks appear at about 0.43V and 1.04V for pH =4, it had only one peak at 0.43V for pH =4.

3.5. Effect of the scan rate

The scan rate was studied at pH (4 and 7), and for step voltage 0.04166V and 0.05951V for each of them, respectively, within the scope of the scan rate (10, 30, 50, 70, 90, 100) mv.s^{-1} (Fig. 6).

From the previous drawing curve, it finds that the scan rate of phenol is 100 (mv.sec^{-1})$^{1/2}$ at pH (7, 4). Under potential-dynamic conditions for an

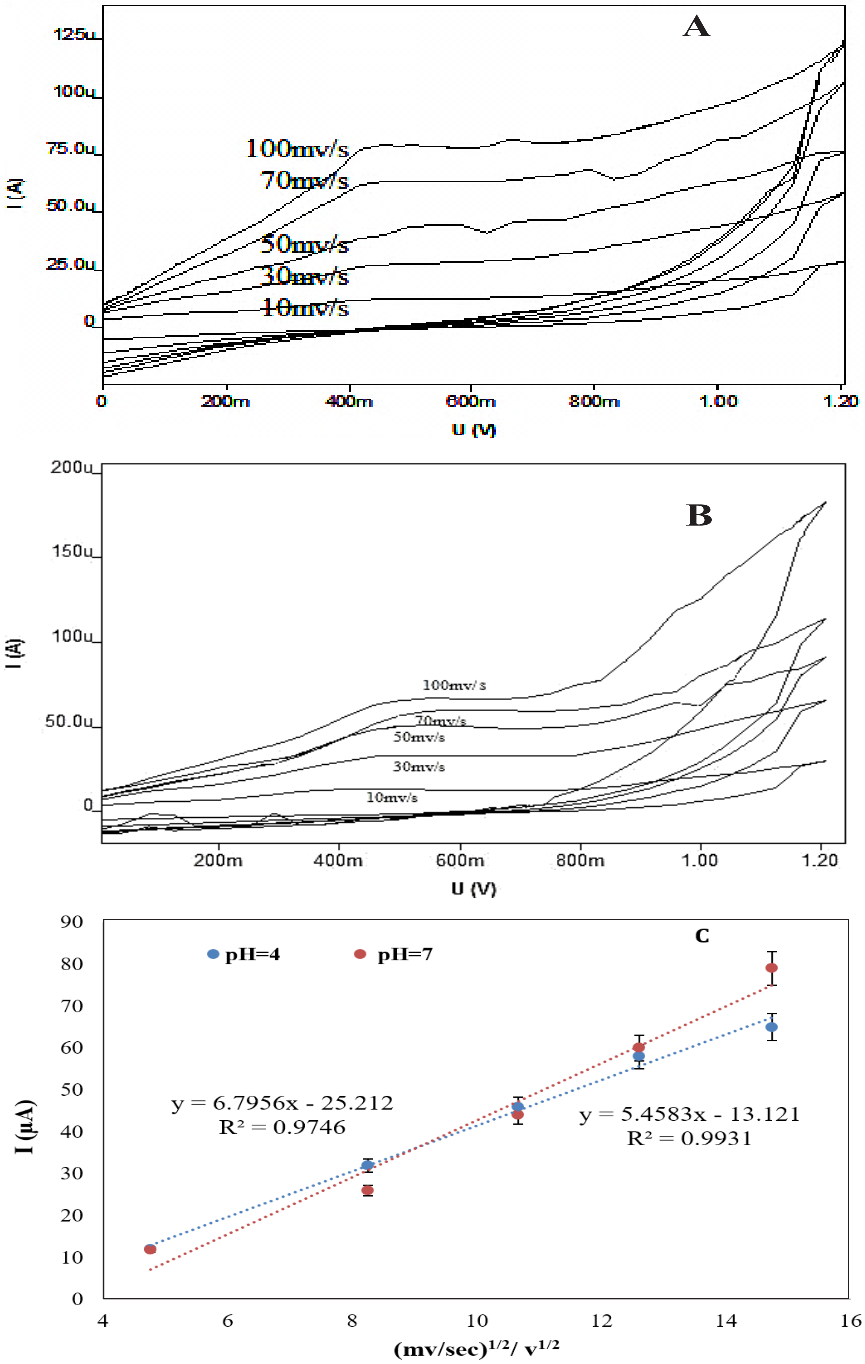


Fig. 6. Effect of scan rates of (10, 30, 50, 70, 90, 100) mv.s^{-1} for 1 mM phenol using KH_2PO4 buffer using the proposed electrode (NiO-NCQD/MCPE):
A) pH= 7; B) pH= 4; C) Effect of scan rates of (mv/sec)$^{1/2}$/ v$^{1/2}$ vs *ip* at pH=(4,7)

irreversible process, the shift in *ip* per a ten-fold increase in scan rate.

3.6. Analytical application

Curves of the phenol concentration are studied to determine the linear dynamic ranges of phenol (10, 250, 500, 750, 1000) µM by (CV) method using a buffer solution of at pH (4,7) using the electrode (NiO-NCQD/MCPE), as shown in Figure 7.

Due to Figure 7, Results clearly showed a linear dynamic range between (10 – 1000) µM with equation $I=0.0428C_{Phenol} + 43.938(R^2=0.9732)$ and

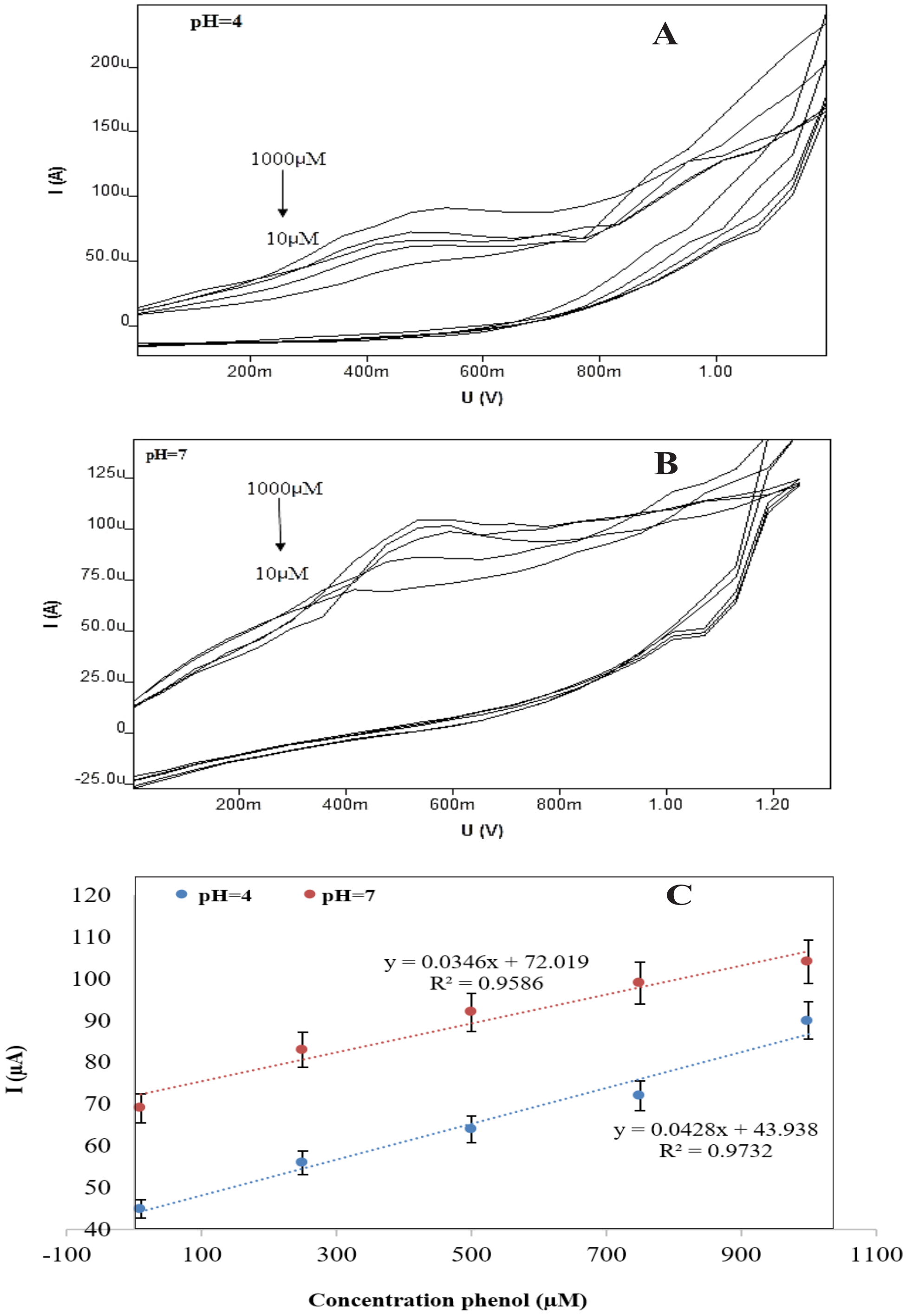


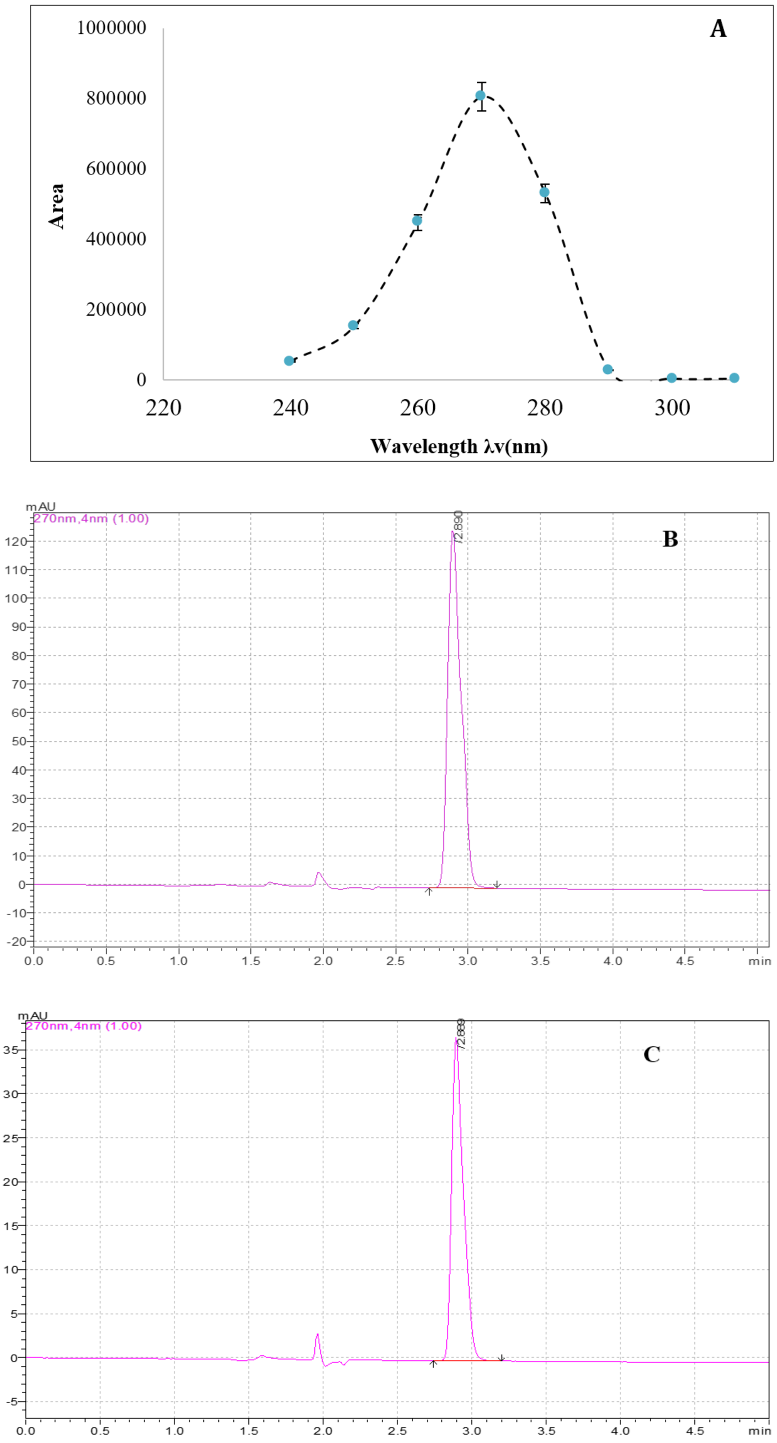
Fig. 7. Curve of phenol concentration (10, 250, 500, 750, 1000) μM vs *ip* by (CV) method using the proposed electrode (NiO-NCQD/MCPE) A: pH=4; B: pH=7, and C: phenol of concentration vs I (μA)

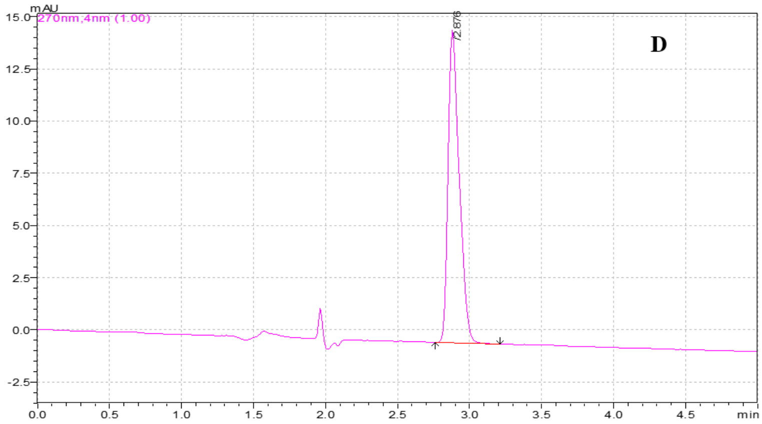
limit of detection (LOD=3SD/m=5.719μM) at pH=4 by (CV) method. The diffusion coefficient (D) is calculated from Randles-Sevcik irreversible. Based on Figure 7, Results clearly showed a linear dynamic range between (10 – 1000) μM with equation I=0.0346C_{Phenol} +72.019 (R^2=0.9588) and limit of detection (LOD=3SD/m=6.391μM) at pH=7 by (CV) method. The diffusion coefficient (D) is calculated from Randles-Sevcik irreversible.

3.7. Phenol analysis using high-performance liquid chromatography HPLC

A standard solution from phenol is prepared at a concentration of 20 mg L^{-1}, then the phenol standard solution is analyzed by HPLC, using a photodiode array detector, in terms of wavelength within a wavelength (240-300) nm. Then, the chromatographic curves were studied, as shown in Figure 8.

From the previous chromatogram curves, it is found that the retention time of the phenol compound was approximately RT=2.982 min, as a graph plotted in terms of wavelength and the area of the chromatographic peaks for the studied solution of phenol, using HPLC in addition to a photo-diode array detector, in terms of wavelength within wavelengths (240-300) nm as in the Figure (8-A).




D
mAU
270nm,4nm (1.00)
2.876
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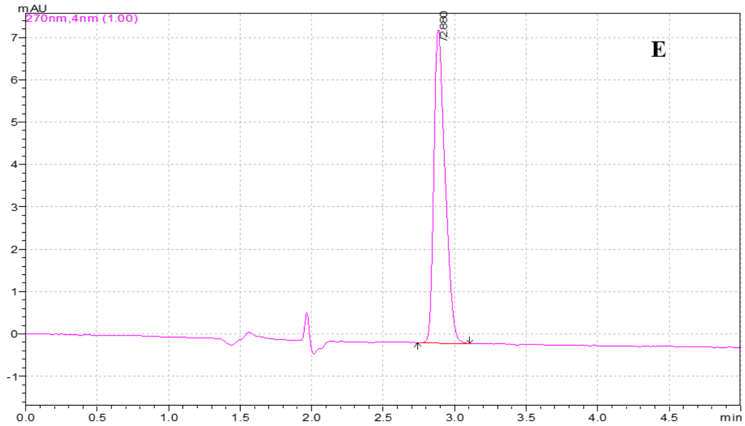
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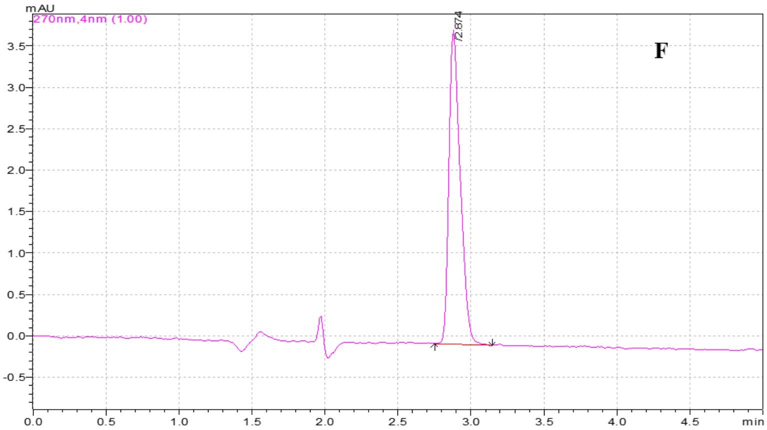
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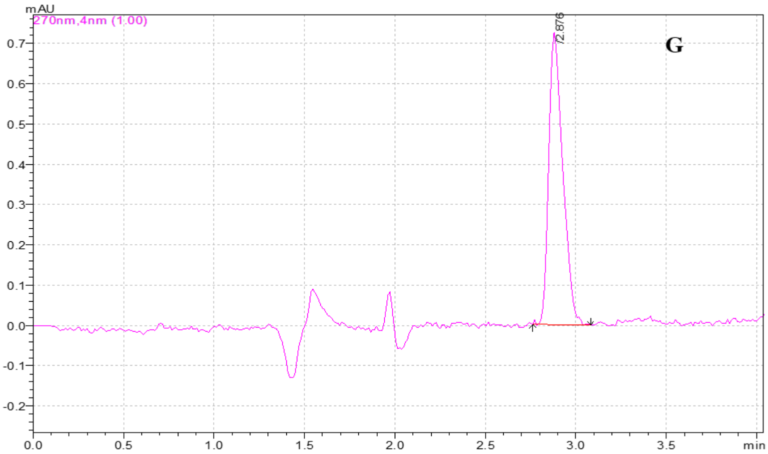
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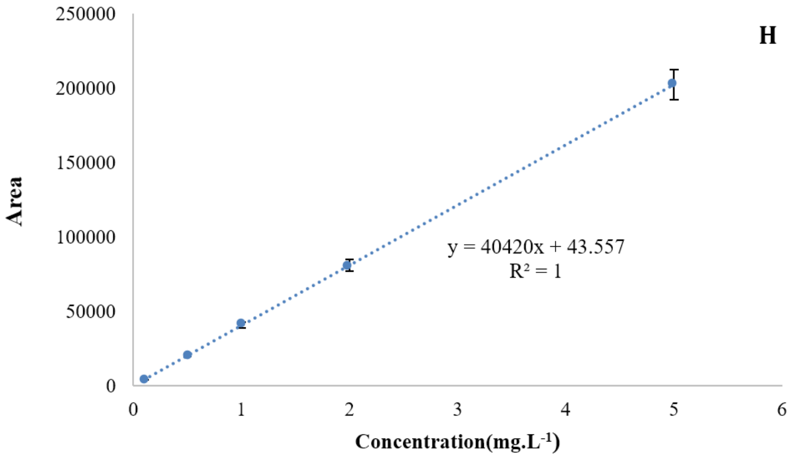

Fig. 8. Determination of phenol-based on high-performance liquid chromatography using a diode photo array detector

A) Graph in terms of wavelength (240-300) nm and area of 20 mg L^{-1}
B) Chromatogram of a 20 mg L^{-1} at 270 nm
C) Chromatogram of a 5 mg L^{-1} at 270 nm
D) Chromatogram of a 2 mg L^{-1} at 270 nm
E) Chromatogram of a 1 mg L^{-1} at 270 nm
F) Chromatogram of a 0.5 mg L^{-1} at 270 nm
G) Chromatogram of a 0.1 mg L^{-1} at 270 nm
H) Curve of series solutions of phenol and chromatographic peak area at 270 nm

It was shown from the previous graph that the best wavelength at which the area has a maximum area is λ (nm) = 270 nm. Accordingly, a series of standard solutions of phenol are prepared with concentrations (0.1, 0.5, 1.0, 2.0, 5.0) mg L^{-1}, then, the relationship between the concentration of standard solutions of phenol was studied in terms of the area of the chromatographic peaks at the time of detention and the previous wavelength, where the graphic curve was drawn as in the Figure (8-H). It was shown from the previous curve that the relationship between the phenol concentration and the area of the chromatography peaks is a direct relationship, $R^2 = 1$, and with the equation: Area = $40420C_{Phenol}$ +43.557, where the series of concentrations falls within the range of (0.1 - 5.0) mg L^{-1}.

3.8. Comparison between the electrode (NiO-NCQD/MCPE) and HPLC

The proposed method modified carbon paste electrode (NiO-NCQD/MCPE) was compared with HPLC to validity and reliability of the method in Table 1. Where, n=3 and at a confidence level of P = 95%, taking into account the value diffusion coefficient, the diffusion coefficient is calculated from Randles-Sevcik for an irreversible system. Through the previous comparison, based on statistical treatments, it is found that the proposed method is reliable. By calculating the Fisher coefficient (F-Test) and comparing it to Fisher's tabular value (F-Tab = 19.000). It was found, depending on the statistical treatments, that the proposed method is characterized by validity and accuracy, as shown by calculating the Fisher F-Test, with a value of (2.219943) and (3.025905) at pH = 4 and pH = 7, respectively. It is found that there is no significant difference between the two methods.

4. Conclusion

This paper deals with fabricating a phenol-selective electrode using carbon paste modified with nickel oxide nanoparticles (NiO) doped with nitrogen

Table 1. Comparison between phenol analysis using modified carbon paste electrode (NiO-NCQD/MCPE) and HPLC

Method	pH	Taken Concentration	Average (mg L^{-1})	SD (mg L^{-1})	RSD٪	R%
NiO-NCQD/ MCPE	4	1.00 mg L^{-1} 10.625 μM	0.992082	0.010506	1.058987	99.1975442
	7		0.984524	0.012266	1.245858	98.4418691
HPLC 270nm	-	1.00 mg L^{-1}	0.989381	0.007051	0.712695	98.93807

carbon quantum dots (NCQD) using cyclic voltammetry. The electrode was manufactured by NIO-NCQD nanoadsorbent. The modified carbon paste consisted of 12% of (NiO - NCQD) and 44% of Graphite powder and 44% of paraffin oil to get a modified carbonate paste. Results best conditions are obtained at pH= 7.0 and 4.0 using KH_2PO_4 buffer. step Voltage and the scan rate are optimized. It found that the scan rate is 100 mv/s, and results clearly showed a linear dynamic range between (10-1000) μM. Then, the phenol standard solution is analyzed by HPLC, using a Diode photo Arrye detector at λ(nm)=270nm, within the range of (0.1-5.0) mg L^{-1}. The proposed method modified carbon paste electrode (NiO-NCQD/ MCPE) was compared with HPLC, it was found that there is no significant difference between the two methods.

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