



## Performance Improvement of Working Electrode Using Grafted Polymer Modified with SiO<sub>2</sub> Nanoparticles

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### Abstract

A new modified glassy carbon electrode (GCE) with grafted polymer (GP)/SiO<sub>2</sub> nanoparticles (SiO<sub>2</sub> NPs) were prepared using mechanical attachment method to produce a new sensor in cyclic voltammetric technique. The new working electrode GP/SiO<sub>2</sub> NPs/GCE was characterized by a standard solution of 1 mM K<sub>3</sub>[Fe(CN)<sub>6</sub>] with 1 M K<sub>2</sub>HPO<sub>4</sub> as an electrolyte to study the redox current peaks of FeII/FeIII ions at different concentrations such as scan rate, pH, determination of diffusion coefficient ( $D_t$ ), reliability and stability of the modified GCE. It was found that the new modified electrode enhanced the redox current peaks of FeII/FeIII from 12  $\mu$ A to 20  $\mu$ A and -5  $\mu$ A to -15  $\mu$ A for oxidation and reduction peaks in GCE, respectively. So, the current ratio ( $I_{pa}/I_{pc}$ ) for the new modified electrode was 1, and the potential peak separation ( $\Delta E_{pa-c}$ ) was 100 mV, which indicated good electrochemical properties as an irreversible electrode and heterogeneous reaction. Good reliability and stability of modified GCE was observed with low detection limit. Scanning electron microscopy (SEM) and atomic force microscopy (AFM) analysis of the nano-deposit was also studied.

**Keywords:** Grafted polymer; Silica nanoparticles; Cyclic voltammetry; FeII/FeIII; GCE

### Introduction

The new study of nanoparticles with different types of polymers is an important subject in the field of electrochemistry, especially in the conversion of insulators into conductive or semi-conductive materials [1-5].

Cyclic voltammetry chronoamperometry, electrochemical impedance spectroscopy and differential pulse voltammetry were used to identify the electrochemical behavior of mitoxantrone at

the sulfonic acid-functionalized SiO<sub>2</sub> nanoparticles (SiO<sub>2</sub> NPs) using glassy carbon electrode (GCE). The determination of mitoxantrone were optimized by the oxidation current peak which was proportional to mitoxantrone concentration in the range of 0.5-173  $\mu$ M, while the detection limit was 36.8  $\mu$ M (S/N = 3) [6]. A modification of grafted polymer (GP) with carbon nanotubes was fabricated as a new working electrode. The working electrode was characterized by K<sub>3</sub>[Fe(CN)<sub>6</sub>] solution in KCl as supporting electrolyte at different concentrations, scan rates and temperatures

using cyclic voltammetric technique [7].

Electrochemical sensors were used for the detection of heavy metals such as lead, cadmium, mercury, arsenic, etc. Stripping voltammetry techniques were applied on electrodes (mercury, bismuth) or electrodes modified at their surface by nanoparticles or nanostructures, carbon nanotube (CNT, graphene). Special attention would be paid to strategies using biomolecules (DNA, peptide or proteins), enzymes or whole cells [8]. A new grafting technique for the functionalized silica particles with anionically produced new polymers was reported. First, the silica nanoparticles were modified with multifunctional chlorosilanes where the original Si-OH surface groups were replaced by Si-Cl groups. Then, the anionically synthesized polymers were linked to the Si-Cl functionalized nanoparticle surface. The polymer linking event was accompanied by termination reactions, most likely due to residual Si-OH groups [9].

Cyclic voltammetry, differential pulse voltammetry and linear sweep voltammetry were used to evaluate some electrochemical aspects of the nanohybrid materials of poly-proline-amino functionalized magnetic mesoporous silica-beta cyclodextrin nanohybrid on GCE [10].

A new nanocomposite based on O-aminophenol (OAP) was prepared by the electropolymerization of OAP at the surface of GCE in the presence of

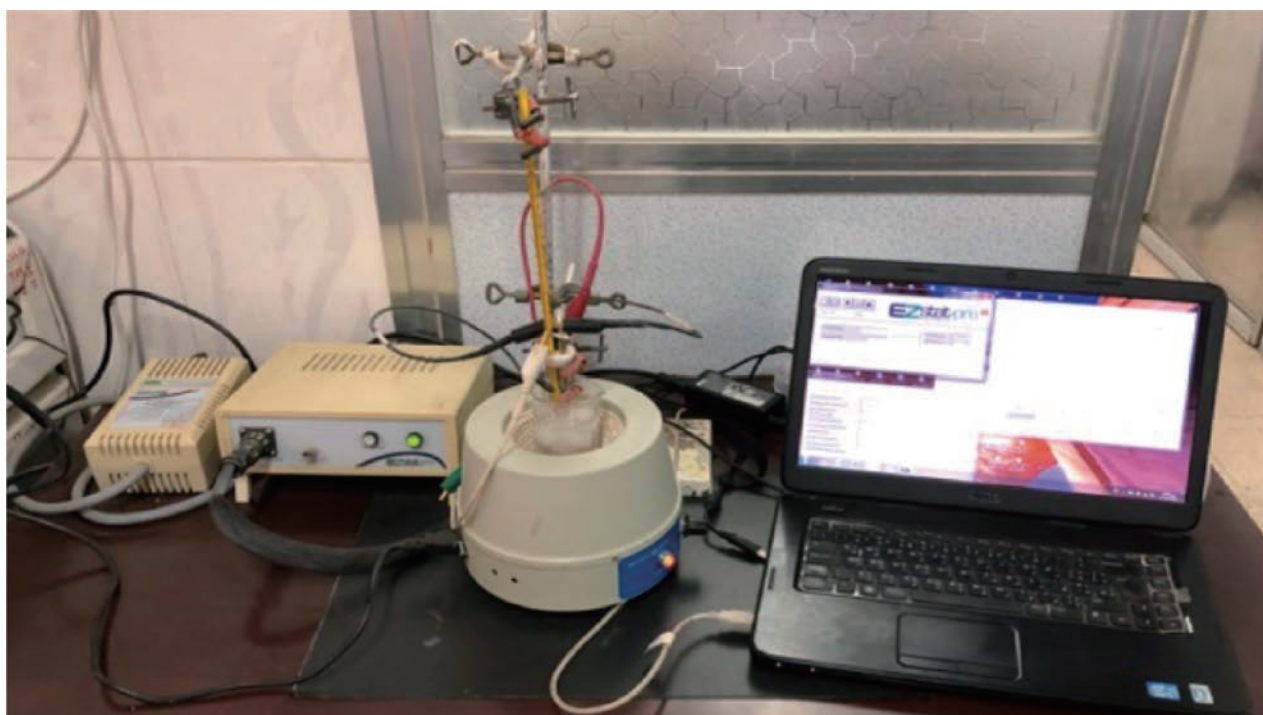
SiO<sub>2</sub> nanoparticles. The cyclic voltammetry and electrochemical impedance spectroscopy (EIS) studies confirmed that the poly O-aminophenol (POAP) nanocomposite films had a higher capacitance than the pure POAP films. The presence of SiO<sub>2</sub> led to an obvious improvement in the overall electrochemical performance of the GCE surface covered by POAP films [11]. CNTs have excellent properties such as small size with larger surface area, high electrical and thermal conductivity, high chemical stability, high mechanical strength, and high specific surface area. They are now used in the fabrication of nanostructured electrochemical sensors, immunosensors and DNA biosensors [12].

In this work, GP was modified with SiO<sub>2</sub> NPs and doping the GCE with these polymer to study in cyclic voltammetric technique.

## Experimental

### Equipments and electro-analytical analysis methods

The potentiostat used in this work was EZstat series (Potentiostat/Galvanostat, NuVant Systems Inc., USA). The cyclic voltammetry experiment was performed by connecting the potentiostat to the electrochemical cell and a computer with special software. The reference electrode was silver/silver chloride (Ag/AgCl in 3 M NaCl) with 1 mm diameter platinum wire as counter



**Fig. 1** Cyclic voltammetry experimental set-up.

electrode. Two modified GCEs were prepared and used as working electrodes. Before using any solutions in the cyclic voltammetric cell, the electrolyte solution was treated with nitrogen gas for 10-15 min to remove oxygen. The cyclic voltammetry cell experimental set up is shown in Fig. 1.

In this work, the surface morphologies and diameter of sample nanoparticles were investigated by scanning electron microscope (SEM) SEM-JEOL operated at 20-30 kV and atomic force spectroscopy (AFM), respectively.

### Procedure

Cyclic voltammetric cell was used in this technique by adding 10 mL of electrolyte in the quartz cell and immersing three electrodes in the electrolyte medium, GP-SiO<sub>2</sub>NPs/GCE as working electrode, Ag/AgCl as reference electrode and platinum wire as counter electrode. Then these three electrodes were connected with potentiostat to find the results by the cyclic voltammogram using personal computer.

### Reagents

Silicon oxide nanoparticles (20-30 nm) was purchased from Hongwu International Group Ltd, China. Potassium ferrous cyanide, dipotassium phosphate and potassium chloride were from Sinopharm Chemical Reagent Co, Ltd. (SCRC), China, Potassium perchlorate and potassium nitrate were from British Drug Houses (BDH), England. The deionized water was used to dilute all the solutions. All materials were with purity of 98-99.9% and were thus used without any further purification process.

### Synthesis of grafted polymer (GP)

By gamma-irradiation technique, polystyrene was grafted with acrylonitrile using chloroform as a solvent and ferrous ammonium sulphate as a catalyst, respectively. Different percentages of the GP were collected and studied [13].

### Preparation of GP/SiO<sub>2</sub> NPs

The GP was first dissolved in chloroform and mixed with nano SiO<sub>2</sub> powder (20-30 nm) by weight ratio of 1000:1 (GP: nano SiO<sub>2</sub>) for 72 h with constant stirring at 50 °C. After the evaporation of chloroform, the precipitate was ground with mortar and pestle into fine particles yield as GP/nanosilica.

### Preparation of modified GCE (GP/SiO<sub>2</sub> NPs/GCE)

The GCE was polished with alumina slurry (0.5

micron) and then ultrasonically cleaned for 10 min, followed by rinsing with distilled water and drying at room temperature (drying by air blower).

The modification of the cleaned GCE with silica nanoparticles was done by mechanical attachment method [14]. The GCE surface was tapped (doping) about thirty times onto GP/SiO<sub>2</sub> NPs powder placed on a filter paper as shown in Fig. 2. In this work, the modified GCE with silica nanoparticles would be used as working electrode and termed as GP/SiO<sub>2</sub> NPs/GCE.

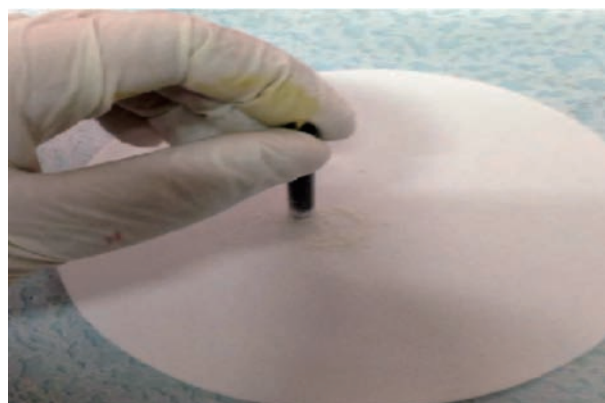


Fig. 2 Mechanical attachment method.

## Results and Discussion

### Characterization of different modified electrode

Fig. 3 illustrates the cyclic voltammogram of different working electrodes (GCE and GP/SiO<sub>2</sub> NPs/GCE) to characterize the potential area of the electrode in 1 M K<sub>2</sub>HPO<sub>4</sub> solution as a supporting electrolyte. A wide potential area of the new modified electrode (GP/SiO<sub>2</sub> NPs/GCE) at -2 to +2 V without any current peaks was found, while the GCE had a range of potential at -1.5 to +1.8 V with current peak at 1-1.8 V. So, the

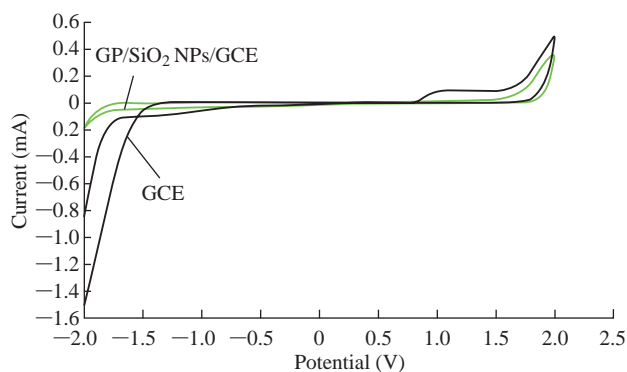
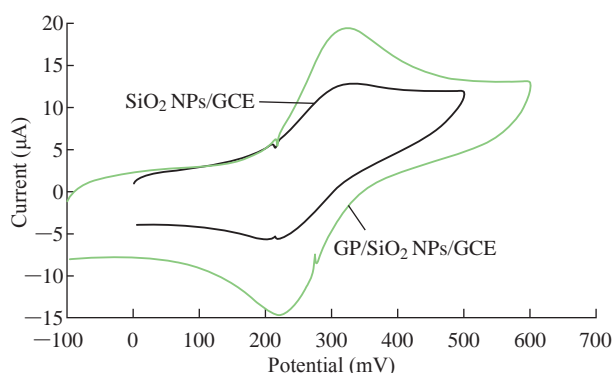


Fig. 3 Cyclic voltammogram of 1 M K<sub>2</sub>HPO<sub>4</sub> using GCE and GP/SiO<sub>2</sub> NPs/GCE as working electrodes versus Ag/AgCl as reference electrode and SR = 100 mV/sec.

modified electrode achieved good electrochemical properties for voltammetric analysis [15].

Normally, in voltammetric analysis, the  $K_4[Fe(CN)_6]$  compound was chosen for standardization and calibration [16] of the new modified electrode GP/SiO<sub>2</sub> NPs/GCE and for comparing it with SiO<sub>2</sub> NPs/GCE as shown in Fig. 4. It was found from the oxidation-reduction current peaks of FeII/FeIII that the modified GCE with SiO<sub>2</sub> NPs had current at 12  $\mu$ A and -5  $\mu$ A respectively, while the current of modified GCE with GP/SiO<sub>2</sub> NPs was enhanced to 20  $\mu$ A and -15  $\mu$ A respectively. This means that GP polymer with nanoparticles acted as an electro-catalyst and increased the conductivity of the modified working GCE. Also, the current ratio of oxidation-reduction current peaks of FeII/FeIII was  $I_{pa}/I_{pc} \approx 1$ . This demonstrates that the new modified electrode GP/SiO<sub>2</sub> NPs/GCE functioned



**Fig. 4** Cyclic voltammogram of 1 mM  $K_4[Fe(CN)_6]$  in 1 M  $K_2HPO_4$  using different modified GCEs as working electrodes versus Ag/AgCl as reference electrode and SR = 100 mV/sec.

as a reversible electrode [17], and the potential peak separation of  $\Delta E_{pa-c} \approx 100$  mV suggests that the reaction at the modified electrode was a homogenous process [18].

### Effect of different electrolytes

The effect of different supporting electrolytes was studied on the oxidation-reduction current peaks of FeII/FeIII ions to find the enhancement of the current on the modified electrode. It was found that the value of enhancement of the oxidation-reduction current in  $K_2HPO_4$  electrolyte was 1.313 and 1.22 respectively, as shown in Table 1. The modified working electrode (GP/SiO<sub>2</sub> NPs/GCE) was more sensitive to the  $K_2HPO_4$  electrolyte in the electro-analysis by cyclic voltammetric technique.

In general, the degree of oxidation current enhancement in varying electrolyte varied in the following order:



Furthermore, the reduction current enhancement was in the following order:



Since  $K_2HPO_4$  produces the highest current output, it was used in the following studies.

### Effect of different concentrations

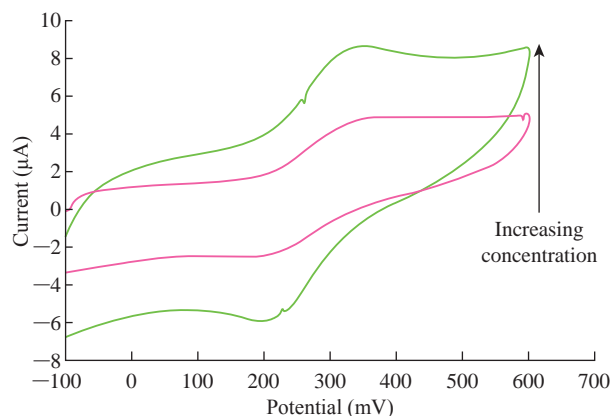
The new modified electrode (GP/SiO<sub>2</sub> NPs/GCE) can be used to find and detect the low concentrations of ions in the aqueous solutions. This electrode is a good

**Table 1** Cyclic voltammetry of 1 mM potassium ferrous cyanide in different electrolytes, 1 M of KCl,  $KNO_3$ ,  $K_2HPO_4$ , NaCl and  $KClO_4$  at scan rate 100 mV/s for the SiO<sub>2</sub> NPs/GCE and GP/SiO<sub>2</sub> NPs/GCE

Electrolytes	$I_{pa}$ ( $\mu$ A) SiO <sub>2</sub> NPs/GCE	$E_{pa}$ (mV) SiO <sub>2</sub> NPs/GCE	$I_{pa}$ ( $\mu$ A) GP/SiO <sub>2</sub> NPs/GCE	$E_{pa}$ (mV) GP/SiO <sub>2</sub> NPs/GCE	Enhancement
Anodic					
KCl	7.36	322	7.71	420	1.047
$KNO_3$	10.6	332	10.7	375	1.009
$K_2HPO_4$	19.8	325	26	364	1.313
$KClO_4$	12.7	298	14.8	320	1.165
NaCl	9.16	363	10.2	384	1.113
Cathodic					
KCl	4.68	200	3.75	126	0.801
$KNO_3$	6.53	179	5.87	150	0.898
$K_2HPO_4$	12.7	201	15.5	179	1.220
$KClO_4$	8.15	136	9.89	126	1.213
NaCl	5.89	160	6.04	155	1.025



sensor for ions by studying the oxidation-reduction current peaks of the  $K_4[Fe(CN)_6]$  compound at low concentrations which enhanced the current against the increasing concentration, as shown in Fig. 5. A

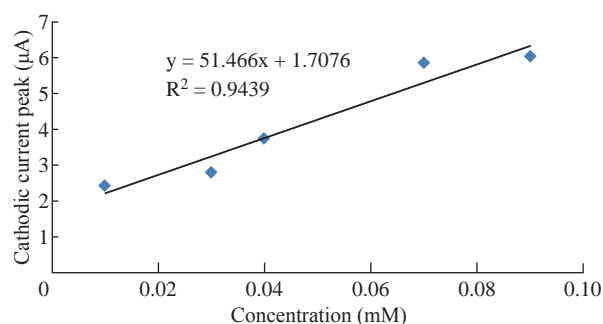


**Fig. 5** Cyclic voltammogram of different concentrations of  $K_4[Fe(CN)_6]$  in 1 M  $K_2HPO_4$  solution on the modified electrode versus Ag/AgCl as reference electrode and scan rate 100 mV/sec.

straight line of the relationship between the oxidation and reduction current was obtained with different low concentrations of  $K_4[Fe(CN)_6]$  with high sensitivity as in Fig. 6 and 7, respectively.

### Effect of different pH

One of the important studies for the characterization of the modified electrode GP/SiO<sub>2</sub> NPs/GCE is

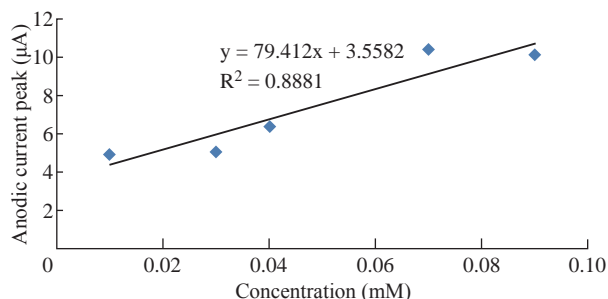


**Fig. 6** Relationship between cathodic current peak against different concentrations of  $K_4[Fe(CN)_6]$ .

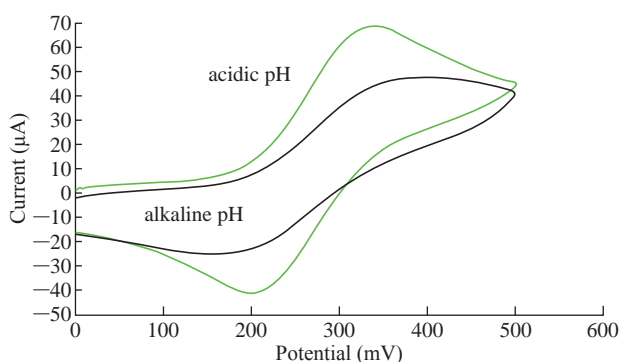
**Table 2** RSD of the stability for modified working electrode (GP/SiO<sub>2</sub> NPs/GCE) of oxidation-reduction current peaks for  $K_4[Fe(CN)_6]$  in 1 M  $K_2HPO_4$

Stability and reliability GP/SiO <sub>2</sub> NPs/GCE				
Anode				
Ipa (X)	X´	(X-X´) <sup>2</sup>	Standard deviation (SD)	Relative standard deviation (RSD)%
6.681	6.6441	0.00136161	0.0289	0.4349
6.68		0.00128881		
6.67		0.00067081		
6.66		0.00025281		
6.65		3.481E-05		
6.64		1.681E-05		
6.63		0.00019881		
6.62		0.00058081		
6.61		0.00116281		
6.6		0.00194481		
Cathode				
Ipc (X)	X´	(X-X´) <sup>2</sup>	Standard deviation (SD)	Relative standard deviation (RSD)%
5.85	5.632	0.047524	0.0103	0.1836
5.8		0.028224		
5.75		0.013924		
5.7		0.004624		
5.65		0.000324		
5.6		0.001024		
5.55		0.006724		
5.52		0.012544		
5.5		0.017424		
5.4		0.053824		

electrochemical property at different pH. Fig. 8 shows the oxidation-reduction current peaks of FeII/FeIII on the new electrode, indicating high current at acidic (pH: 2-6) and low value at alkaline medium (pH: 8-12). Thus, the modified working electrode acted in acidic medium as electro-catalyst; also, the modified electrode could be used in acidic and basic media [19].



**Fig. 7** Relationship between anodic current peak against different concentrations of  $K_4[Fe(CN)_6]$ .



**Fig. 8** Cyclic voltammogram of  $K_4[Fe(CN)_6]$  in  $K_2HPO_4$  solution at acidic and alkaline pH on GP/SiO<sub>2</sub>NPs/GCE versus Ag/AgCl as reference electrode.

## Reliability and stability of the electrode

The stability of modified materials on the GCE was studied for ten times of oxidation-reduction current peaks of  $K_4[Fe(CN)_6]$  in  $K_2HPO_4$  solution as shown in Fig. 9. The relative standard deviation (RSD) of anodic and cathodic peaks was determined with good value as of  $\pm 0.4349$  and  $\pm 0.1836$ , respectively (Table 2).

## Effect of different scan rates

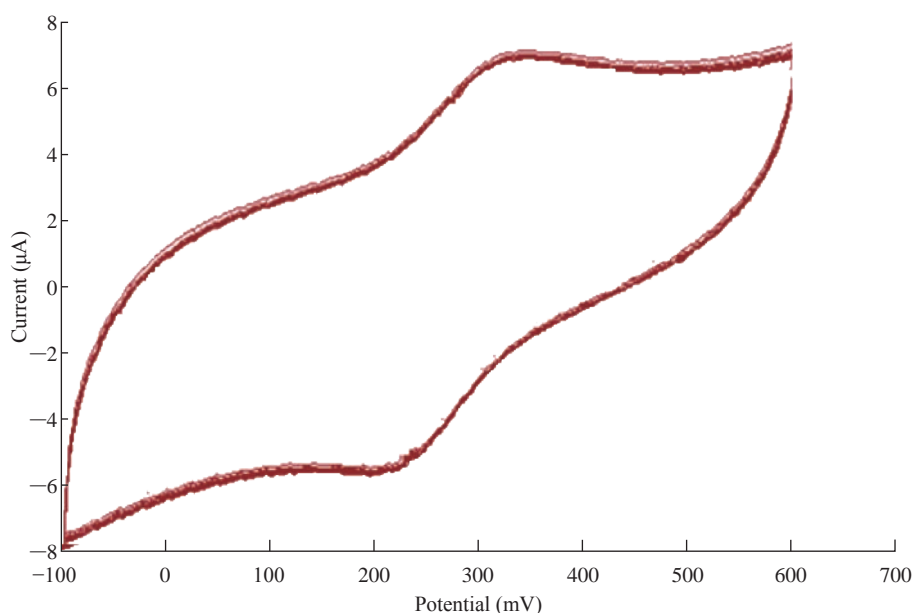
Different scan rates from 0.01 to 0.1 V/sec were studied for the oxidation-reduction current peaks of FeII/FeIII in 1 M  $K_2HPO_4$  solution as an electrolyte on the modified electrode (GP/SiO<sub>2</sub>NPs/GCE) as shown in Fig. 10. It was found that the redox peaks of FeII/FeIII were enhanced with increasing the scan rate. Hence, the new modified electrode acted as electro-catalyst with the presence of silica nanoparticles in the structure of GP.

Diffusion coefficient value was determined from the Randles-Sevcik equation which describes it as reversible redox couple peaks [20, 21]:

$$I_p = (2.69 \times 10^5) n^{3/2} A C D_f^{1/2} V^{1/2}, \quad (1)$$

where  $I_p$  is the current peak ( $\mu A$ ),  $n$  is the number of moles of electrons transferred in the reaction,  $A$  is the area of the electrode ( $cm^2$ ),  $D_f$  is the diffusion coefficient ( $cm^2/sec$ ), and  $V$  is the scan rate of the applied potential (V/sec).

The diffusion coefficient values of oxidation-reduction reaction for FeII/FeIII ions in  $K_2HPO_4$



**Fig. 9** Cyclic voltammogram of FeII/FeIII current peaks on the modified GCE at ten times of scan rate versus Ag/AgCl as reference electrode.

solution on GP/SiO<sub>2</sub> NPs/GCE was determined as  $D_{fa} = 6.03 \times 10^{-6} \text{ cm}^2/\text{ec}$  and  $D_{fc} = 3.122 \times 10^{-6} \text{ cm}^2/\text{sec}$ , respectively.

### Scanning electron microscopy (SEM) study

Fig. 11 illustrates the SEM of the GP structure with silica nanoparticles. The silica nanoparticles were incorporated inside the GP structure [22-26].

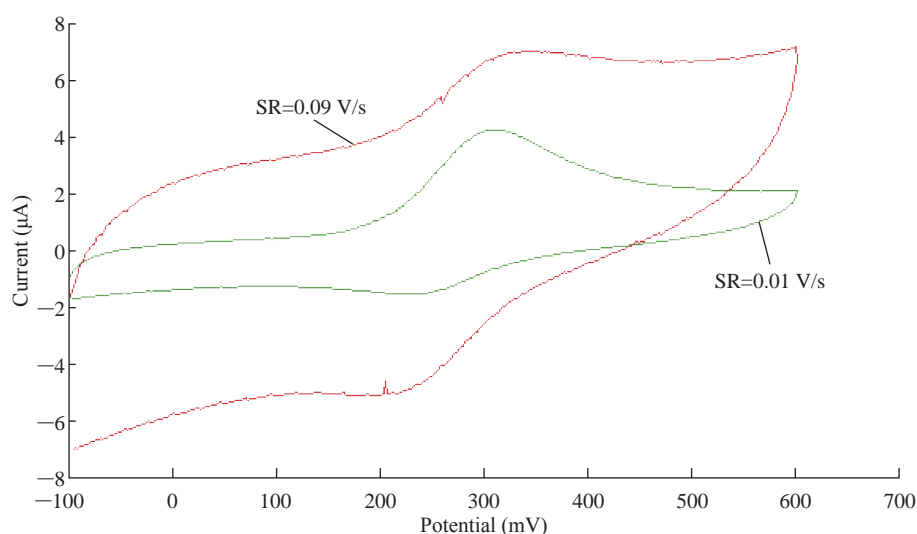
### Atomic force microscopy (AFM) study

The AFM image of the GP material modified with silica nanoparticles GP/SiO<sub>2</sub> NPs is shown in Fig. 12. The average SiO<sub>2</sub> NPs diameter was 50 nm as shown in

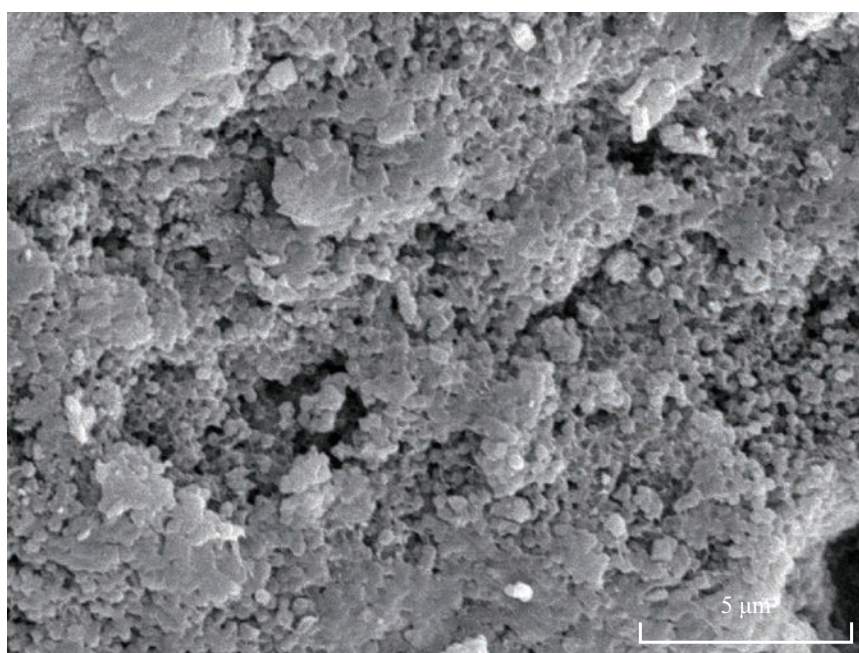
Fig. 13.

## Conclusions

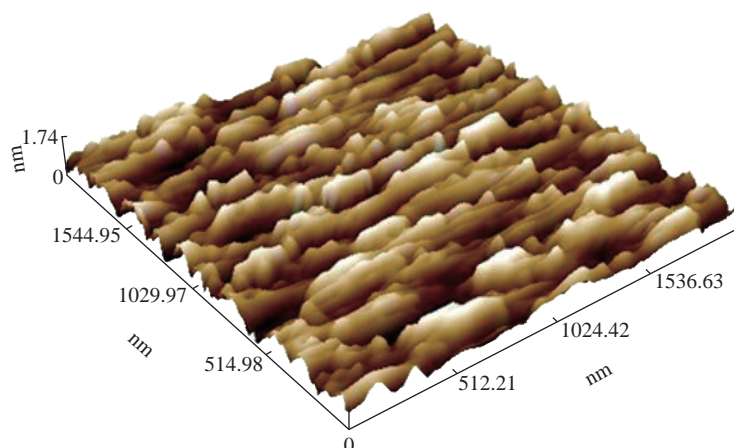
In this study, high sensitivity to low detection limit of concentration in aqueous solution, good reliability with stability on the electrode and resistance to different pH were obtained using new working electrode GP/SiO<sub>2</sub> NPs/GCE. The diffusion coefficient of the redox current peaks from different scan rates of FeII/FeIII was determined by using Randles-Seveik equation. And SEM and AFM images confirmed the



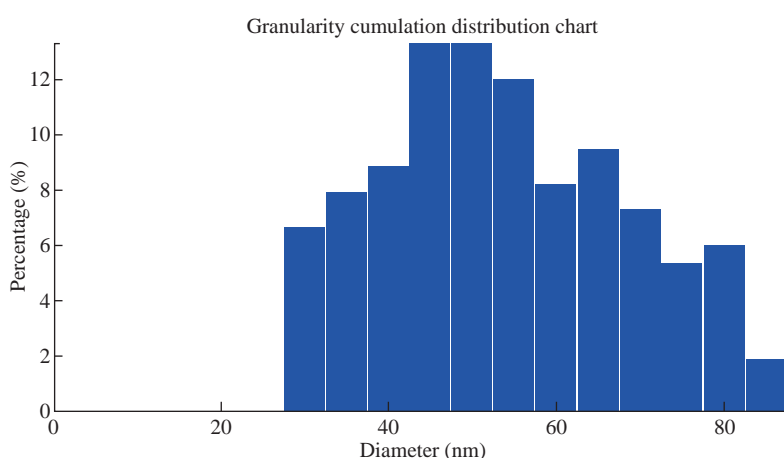
**Fig. 10** Cyclic voltammogram of oxidation-reduction for FeII/FeIII in K<sub>2</sub>HPO<sub>4</sub> solution on GP/SiO<sub>2</sub> NPs/GCE at different scan rates of 0.01-0.1 V/sec.



**Fig. 11** SEM of GP/SiO<sub>2</sub> NPs.



**Fig. 12** AFM of GP/SiO<sub>2</sub> NPs.



**Fig. 13** Scanning prob microscop (SPM) of GP/SiO<sub>2</sub> NPs.

nanoparticle structure of the GP.

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## Conflict of Interests

The authors declare that no competing interest exists.

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