A Novel Method for Ultra Trace Measurement of Bentazon Based on Nanocomposite Electrode and Continuous Coulometric FFT Cyclic Voltammetry

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ABSTRACT: In this work, a novel electrochemical technique is presented for determination of Bentazon using a nanocomposite based sensor and Continuous Coulometric Fast Fourier transformation cyclic voltammetry (CCFFTCV). In this method, the sensor response is in coulomb and is calculated based on integrating charge changes under the current voltammogram in selected potential range. The new sensor was constructed using MWCNT mixed with ionic liquid and reduced graphene oxide on a SiC NPs modified carbon ionic liquid electrode. The experimental conditions were then optimized. The modified electrode has good electrocatalytic activity toward the oxidation of Bentazon. The presence of the nanoparticles enhanced the electrochemical signal which increased the sensitivity of the sensor. Moreover, the proposed sensor exhibits good accuracy, the response time less than 7s, with good repeatability (R.S.D value of 2.3%) and long term stability. The linear concentrations range of Bentazon was from 1–150 nM with a detection limit of $2.5\pm0.01\times10^{-10}$ M.

Key words: Bentazon, FFT cyclic Voltammetry, MWCNT, Reduced Graphene oxide

INTRODUCTION

Bentazon (Bentazone, benazolpine) 3-(1-methylethyl)-1-H-2,1,3-benzothiadiazin-4(3H)-one 2,2-dioxide (Fig. 1) belongs to the thiodiazine family (Fan, 1999). Now, pesticide (like Bentazone) pollution is the main environmental subject for areas close to the sedges in beans, rice, corn, peanuts and mint growing areas, since pesticides in those field are easily discharged to the open environment. Several analytical techniques are used for the determination of Bentazon in water, soils and fruits such as gas chromatography (Cessna, 2002) and high performance liquid chromatography (HPLC) (Pinto and Jardim, 1999). However, the electrochemical oxidation properties of Bentazon were studied and an electrochemical method was developed for determination of Bentazon in a commercial product (Garrido, 1998; Akinbulu, *et al.* 2009; Rahemi, *et al.* 2013). Different forms of carbon nanostructures, such as multiwall carbon nanotubes (MWCNTs), reduced graphene oxide nanosheets (RGO) and Silicon Carbide (SiC) Nanoparticles (NPs) demonstrate high surface area in new designed sensors (Norouzi, *et al.* 2011; 2012a,b; 2013a,b). Furthermore,

existing the large area of the edge-plane sites on RGO and MWCNTs provide a condition for rapid heterogeneous electron transfer of redox compound. Another way to enhance the performance of sensors is application of ionic liquids (IL) in modifier composition which would be very helpful. An example of its application is in construction of the carbon paste electrodes, which are called carbon ionic liquid electrodes (CILEs). These electrodes have established specific characteristics, including increased reversibility, higher sensitivity, wider electrochemical windows and the ability to lower the overpotentials of electroactive compounds. Accordingly, it is expected that using IL that casted on RGO and MWCNTs, exhibited catalytic activity towards redox processes of compounds.

In this work, a new electrochemical method is introduced for determination of Bentazon. The coulometric FFT cyclic voltammetry (CCFFTCV) technique (Norouzi, *et al.* 2005a,b,c; 2006a,b; 2007a,b; 2008a,b,c; 2014; Alizadeh, *et al.* 2010) combined with a new modified electrode was used for sensitive detection of Bentazon. The electrode was designed

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based on IL-MWCNT and RGO hybrid on CILE modified with SiC nanoparticles. The presence of SiC NPs and RGO in modification of the electrode provides an environment which enhances the electrocatalytic activities. Scanning electron microscopy and impedance spectroscopy was used to characterize the electrode surface. Under optimal conditions, the proposed sensor exhibited a linear response to a wide concentration rage of Bentazon.

MATERIALS & METHODS

Bentazon, potassium ferricyanide, sodium chloride, potassium chloride, sodium phosphate dibasic (Na_2HPO_4), sulfuric acid (98%), ethanol (98%) were all purchased form Merck Co. 1-butyl-3-methylimidazolium tetrafluoroborate (BMIM-BF_4, ionic liquid, IL) were of analytical grades all from Merck Co. Silicon carbide (SiC) powders with average particle size of 10-25 nm, supplied by IROC Corporation, Iran. Buffer solutions 0.05 M (prepared by H_3PO_4 and NaOH) at pH 4.5 was used as the supporting electrolyte. The prepared solutions were kept at 4 °C before use.

CILE was prepared by hand-mixing 5 g of graphite powder and 2 g of IL in a mortar and heated to form a homogeneous carbon paste. The paste was packed into a cavity of Teflon tube (i.d. 4 mm). An electrical contact was established via a copper wire to the paste in the inner hole of the tube. It was then left to cool to room temperature. A mirror-like surface was obtained by smoothing the electrode onto a weighing paper.

Graphene nanosheets oxide (GO) was prepared by a modified Hummers method using expandable graphite flake as the original material (Sun, *et al*. 2008). After that, chemical reduction of the 1.0 mg/ml GO powder was done in distilled water with 150 mM $NaBH_4$ (Wojcik and Kamat, 2010). The mixture was kept at 90 °C for 30 min. The product was centrifuged to precipitate the RGO powder, which was eventually dried in vacuum at low temperature. SiC NPs was added to 10 ml of ethanol to obtain (1.0 mg/ml). The mixture was ultrasonicated for another 30 min. To obtain SiC/CILE, 10-50 µl of ethanol SiC solutions was casted on the surface of CILE. To obtain RGO/SiC/CILE, then, RGO was added to the methanol (to produce 1.5 mg/mL of RGO) and ultrasonicated for 30 min. Then 10-80.0 µL of RGO mixture solution was casted on the surface of SiC/CILE and left it to dry at room temperature to form a stable film. Then, 10 mg of MWCNT added to 10 ml IL and ultrasonicated for 30 min. Then, to obtain MWCNT-IL/RGO/SiC/CILE with a microinjector 5-80 µL of MWCNT-IL mixture solution was casted on the surface of RGO/SiC/CILE and left it to dry at room temperature to form a stable film. The prepared sensor was stored at 4 °C in PBS before use. The schematic diagram of the steps of construction of the Bentazon sensor is shown in Fig. 2.

For CCFFTCV voltammetric measurements a homemade potentiostat was used which was connected to a PC. The computer equipped with an analog to digital data acquisition board (PCL-818H, Advantech
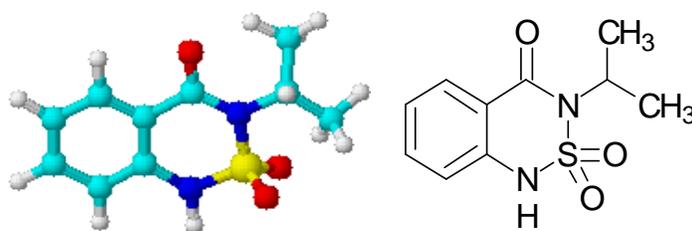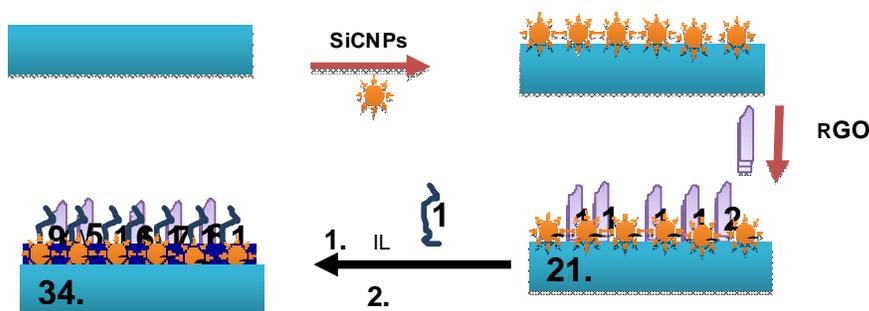

Fig. 1. Structure of Bentazon

Fig. 2. Schematic steps of the electrode preparation

Co.). Electrochemical software was developed using Delphi 6.0 for applying the potential, data acquisition, processing and plot the data in real time.

The program, also, was used to generate an analog waveform and acquire current readings. The potential waveform was repeatedly applied to the working electrode and then the data was acquired, and stored by the software.

RESULTS & DISCUSSION

Fig. 3 shows the response of MWCNT-IL/RGO/SiC/CILE (curves a to f) to 1.2×10^{-6} M of Bentazon in 0.05 M phosphate buffer solution at pH 4.5, at scan rate of 100 to 1600 mV/s, and the inset curve in Fig. 3 shows the CVs of Bentazon on the bare CILE. A low redox activity is observed at the unmodified CILE electrode over the applied potential range. As shown, at the bare electrode Bentazon shows a very weak anodic peak which is an indication of the weaker adsorption reaction of Bentazon on the electrode surface. Conversely, for MWCNT-IL/RGO/SiC/CILE electrode, there is a well-defined anodic and a small cathodic peak in the voltammogram, at potentials 780 and 170 mV, respectively.

Moreover, a considerable negative shift of the anodic peak potential and remarkable increase of current is indicating significant catalytic ability of nanoparticles to oxidation of Bentazon. This could be due to enhancement of the rate of electron-transfer and make more accumulation Bentazon molecules. The current improvement could also be due to the existing a lager surface area and conductivity of the composite.

The oxidation peak currents rise linearly with the linear egression equations as $i_p = 3.2v^{1/2} +0.29$ ($R = 0.983$), suggesting that the reaction is diffusion-controlled behavior with an electron transfer process. It can be seen that with the increase of the scan rate, the oxidation peak currents increases with the same rate. Fig. 4 shows SEM image of the surface of the constructed MWCNT-IL/RGO/SiC/CILE. In this Fig., it can be seen the distribution of SiC RGO and typical crumpled structure. In fact, the composite surface is well-coated with RGO and SiC the diameters 30–60 nm. This is reasonable to deduce that MWCNT-IL played an important role here. As suggested by other researchers, the conductance of IL is higher at surface due to presence a low interfacial tension. This also can enhance current, as well as, the sensitivity of the sensor.
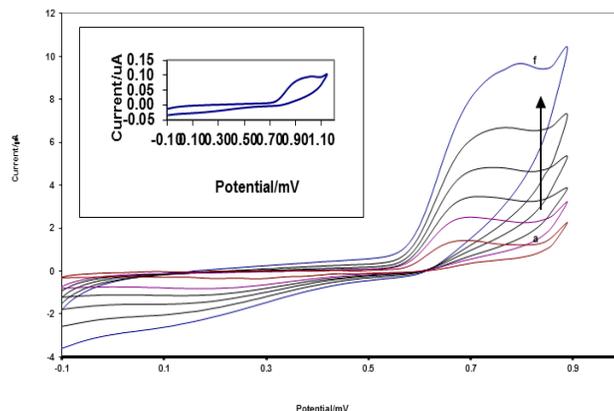

Fig. 3. Typical cyclic voltammograms of MWCNT-IL/RGO/SiC/CILE at different scan rates, 0.1, 0.2, 0.4, 0.6, 0.8, 1.6 V/s (a to f) in 1.2×10^{-6} M Bentazon in 0.05 M PBS at pH 4.5
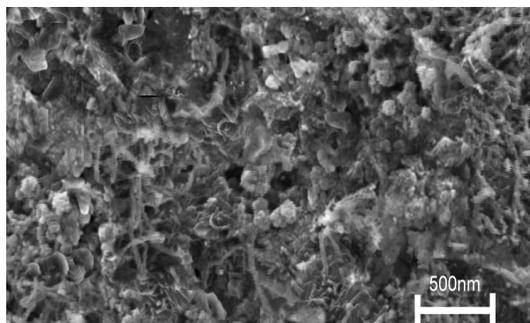

Fig. 4. SEM images of the surface of MWCNT-IL/RGO/SiC/CILE

Fig. 5A shows CCFFTCV voltammogramms and the changes in voltammetric response of MWCNT-IL/RGO/SiC/CILE, in 3.0×10^{-7} M Bentazon 0.05 M PB solution at pH 4.5, the potential range of -100 to 900 mV, and at scan rate of 2.0 V/s. In this graph, the time axis represents the time window of the experiment run (Norouzi, *et al*. 2011; 2012 a,b; 2013a,b).

The Fig. shows that there is no current for the sensor in absent of the analyte (due to background subtraction), but after spiking of Bentazon a considerable response current appears at potential of 780 mV. However, as mentioned above the accumulation of Bentazon to high surface area of the sensor can enhance the direct electron transfer between the active sites of the sensor. This can increase the current at the recorded voltammograms when the Bentazon sample was spiked. In this detection technique, MWCNT-IL/RGO/SiC/CILE response was based on the charge under peak in the recorded CVs. The unit for the resulted signal is change from ampere to coulomb (C), which is the charge changes (ΔQ) under the CV curve at a selected

potential range, E_1 to E_2. The equation for sensor response calculated as follows:

$$\Delta Q_n = Q_n - Q_{ave} \text{ for n} > 0 \tag{1}$$

or

$$\Delta Q_n = \int_{E_1}^{E_2} \Delta i_{(n,E)} dE - ave\left[\int_{E_1}^{E_2} \Delta i_{(m,E)} dE\right] \tag{2}$$

where Q_{ave} and Q_n are the calculated average charges at the selected potential range, E_1 to E_2, from m CVs and the calculated charge at the same potential range from subsequent n^{th} cyclic voltammogram, respectively.

Fig. 5 shows the calculated sensor response to spiking of standard solutions of 3×10^{-7}M Bentazon in PB solution at pH4.5. The potential range was the integration range for the ÄQ is 100 to 800 mV. To get the best performance of the detector, the effect the most important experimental parameters were examined and optimized.
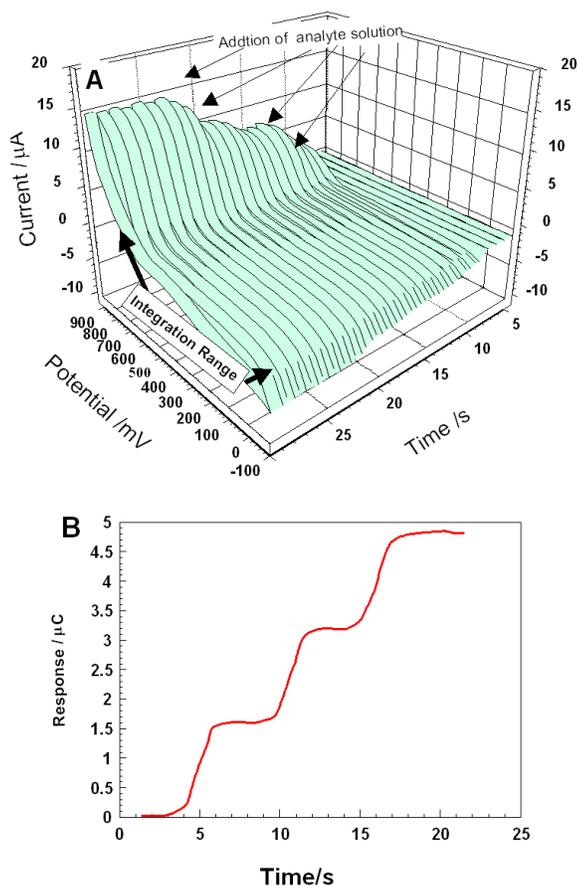

Fig. 5. A) CCFFTCV voltammograms of MWCNT-IL/RGO/SiC/CILE in absent and spiking of the standard solutions of 3.0×10^{-7} M Bentazon in PB solution at pH4.5. The potential range was of -100 to 900 mV at potential scan rate 2.0 V/s. B) The calculated response for sensor in integration range of 100 to 800 mV
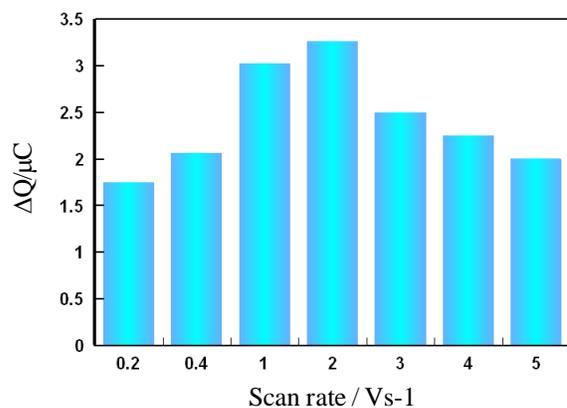
Fig. 6. The effect of the scan rate on the response of the MWCNT-IL/RGO/SiC/CILE to standard solutions of 2.0×10⁻⁷ M Bentazon in PB solution at pH 4.5. The potential range was of -100 to 900 mV, at integration range of 100 to 800
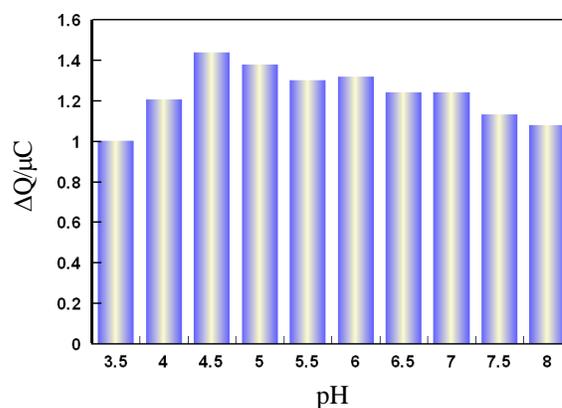

Fig. 7. The effect of pH concentration on sensor response to 2.0 ×10⁻⁷ M Bentazon in 0.05 M PB solution. The potential range of integration is 100 to 800 mV, at potential scan rate 2 V/s
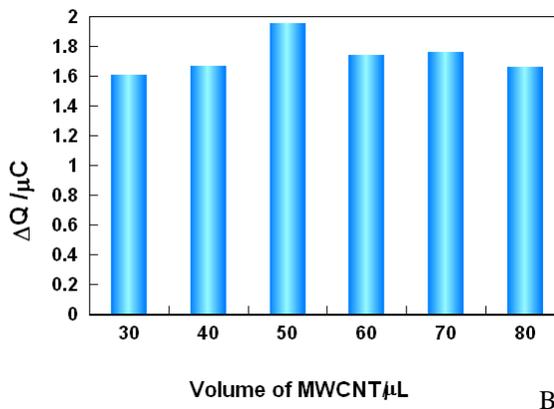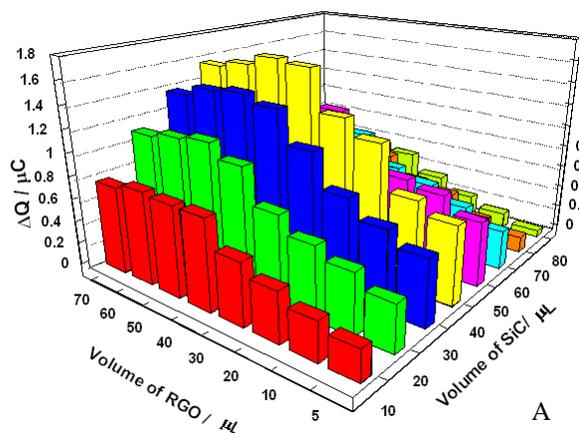

Fig. 8. A) The effect of amount of RGNS, SiC and B) MWCNT, the on the response of the sensor to 2.0×10⁻⁷ M of Bentazon in 0.05 M PB solution at pH=4.5. The potential range of integration is 100 to 800 mV, at potential scan rate 2 V/s

Due to this fact that the sensitivity of the new detection system principally depends on the potential scan rate, it is necessary to optimize the scan rate. Consequently, the change of the sensor response to addition of a solution $5.0×10^{-7}$ M of Bentazon, at scan rates 0.1 to 5 V/s) with the potential was investigated. As it is clear from the Fig. 6, the sensor exhibits the maximum sensitivity at scan rate 2 V/s. The effects of the scan rate on the sensor response could be due to role of kinetic factor of electrochemical processes in the development of peak current. However, it seems that at higher scan rates the noise level of the measurements increased, as well as the current vale. This could be main reason for limitation of using scan rates higher than 2 V/s. Besides, the effects of the potential scan rate on the detection performance can be taken into consideration from different aspects, such

as the speed in data acquisition during the current sampling, which may owe limitation in conversion of the analog-to-digital broad, and rates of electrochemical processes.

The dependence of the electrochemical method on the solution pH was studied over the range pH 3.5 to 8.0. Fig. 7 shows the changes of DQ (or sensitivity) for the oxidation current at recorded at scan rate of 2 V/s when the sensor was subject to $2.0×10^{-7}$ M Bentazon solution at various pH of PBS. The peak potentials and peak currents were dependent on pH values of solution. The peak potentials for reduction of all bases are negatively shifted with increasing pH values.

The results showed that at the pH values less than 4.5, the peak current increase smoothly with pH, up to

Table 1. The comparison of the proposed sensor with the best previously reported ones based on the utilization of different materials

Ref.	Detection Method	DL	Materials
Garrido, 1998	Differential pulse and square wave voltammetry	$100 \, \mu M$	glassy carbon electrode
Akinbulu, *et al.* 2009	Amperometry	$2.48 \times 10^{-7} \, M$	polymeric film of a new manganese phthalocyanine complex octa-substituted with 2-diethylaminoethanethiol
Rahemi, *et al.* 2013	voltammetry	$1.6 \, \mu M$	β-CD/MWCNT modified GC electrode
This work	CCFFTCV	$0.25 \, nM$	MWCNT-IL/RGO/SiC/CILE

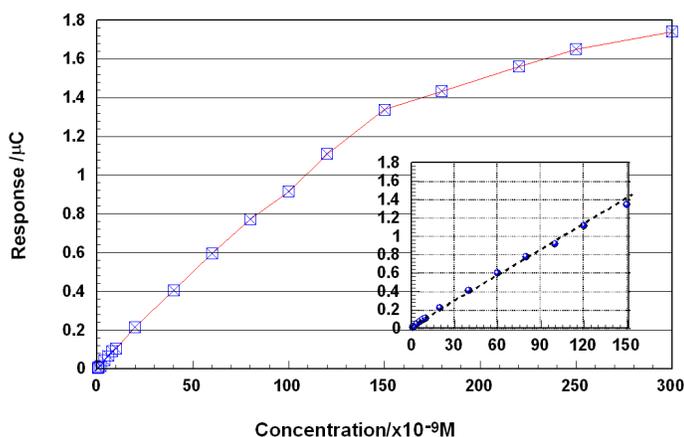**Fig. 9. The calibration curve for Bentazon determination; the inset, response of the sensor to Bentazon upon the following concentrations: 1 to 300 ×10⁻⁹ M PB solution, pH4.5, the potential range of integration is 100 to 800 mV, at potential scan rate 2 V/s**

pH 4.5. After that, at higher pHs the response decreased. Nevertheless, form the graph, it can be concluded the best pH for the sensor operation is around 4.5. It can be expected that amount of SiC NPs, MWCNT and RGO, could change the performance of MWCNT-IL/RGO/SiC/CILE due to change in surface area, conductivity and morphology of the compostie. The relationship between the amount of SiC NPs and RGO in the solution of them was investigated, in which constant volume of those solutions were casted on the electrode surface. Fig. 8A shows the dependence of the sensor response, at potential scan rate of 2 V/s, to volume of RGO and SiC. The sensor was tested to addition of $2.0 \times 10^{-7} M$ Bentazon in 0.05 M PBS at pH 4.5. As shown in the Fig., the value of $ÄQ$ increase with the weight of RGNS reaches to up to 60 µL the sensor response set at the maximum value. Whiles, at the higher amount of RGNS the value of $ÄQ$ decreases, which could be due to filling the holes in the surface and lessening the surface interaction or activity of SiC NPs. Moreover, the change in the sensor response with the amount of MWCNT in the content of the modifier was investigated. Fig. 8B shows the change of the sensor response with the amount of casted

MWCNT. As shown in the Fig., the sensor response initially increases with volume of the casted MWCNT, up to 50 µL, and after that value the response slightly decreases. Therefore, the optimum volume for deposition for MWCNT was 50 µL. It seems that at higher volume of MWCNT the existing species on the RGO/SiC/CILE composite and the active surface area lessening. As mentioned above, the value of the sensor response depends on the choice of the potential integration range in the CCFFTCV measurements. Accordingly, the sensor response to Bentazon sample solution was in µC, where the experimental parameters were set at optimum values in order to obtain the best detection limits for the electrochemical measurement technique. The inset cure in Fig. 9 illustrates a typical calibration graph of the sensor response $ÄQ$) for a set of standard solutions of Bentazon (from 1.0 to300 nM in PB solution at pH=4.5). In this Fig., each point represents the integrated signal for 3 consecutive additions of the Bentazon standard solution. In general, the response of the analyte showed a linear dynamic range of 1 to 150 nM (the inset graph in Fig. 9), A correlation coefficient of R=0.997 values. The detection limit, estimated based on signal to noise ratio

(S/N=3), was found to be $2.5\pm0.01\times10^{-10}$ M. The sensitivity retained 93.4% of initial sensitivity up to 90 days. It was gradually decline, which might be due to the loss of the catalytic activity.

In evaluation, the performances of the fabricated sensor is compared with some of the best previously reported Bentazon sensors based on the utilization of different materials as the working electrode and different detection techniques (Table 1). This result confirms that the presented MWCNT, RGO and SiC NPs based Bentazon sensor with CCFFTCV exhibited an excellent sensitivity for the detection technique.

CONCLUSIONS

This paper presents a highly sensitive electro-chemical detection method for determination of Bentazon. In this method, the sensor response is in coulomb and is calculated based on integrating charge changes under the current voltammogram in selected potential range. Application of in constructed sensor provides high surface area for adsorption along with facilitation of electrode transfere. The reproducibility of MWCNT-IL/RGO/SiC/CILE electrode for catalytic oxidation of Bentazon is evaluated by five independently prepared modified electrodes. Reproducible sensitivity response time less than 7 s and detection limit of 0.25 nM was observed sensor (R.S.D value of 2.3%). The long-term storage stability of the sensor was tested for 90 days.

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