# Paracetamol Sensing Via Modified Electrodes Based On Nano-Structured Bi(III) Schiff Base–Nio Composites

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#### Abstract

In this study, the developed a series of nano-structured Bi(III) Schiff base complexes and their NiO-based hybrids for detecting paracetamol (PA) using electrochemical methods. Modified glassy carbon electrodes (GCEs) were tested using cyclic voltammetric (CV), differential pulse voltammetric (DPV) and square wave voltammetric (SWV) at pH 1.0 under ion beam irradiation (25 to 100 mV/s). Among them, the NiO-Nitro Schiff base Bi(III) composite (NiO-Nitro SBC) showed the best performance, delivering a DPV peak current of 67.8  $\mu$ A at potential 0.59 V nearly ten times higher than the bare GCE. It also showed excellent sensitivity (0.052  $\mu$ A/ $\mu$ M), a low detection limit (0.29  $\mu$ M), and strong linearity (R² = 0.9993). results supported these findings with a peak current of 57.6  $\mu$ A and R² = 0.9998. These improvements stem from the synergistic effects of NiO, redox-active Bi (III), and nitro groups. Overall, the modified electrode offers a promising platform for sensitive and reliable paracetamol detection.

Keywords: Paracetamol; Schiff base complex; Bismuth(III); NiO nanoparticles;; Differential pulse voltammetry;

#### **INTRODUCTION**

Paracetamol is widely used to relieve pain, and can cause liver damage in excessive amounts so precise measurement is important in both medicine and quality control of drugs [1, 2]. Conventional procedures are effective but slow and costly [3,4]. On the other hands, electrochemical detection is easy to operate and economic, and has high sensitivity [5]. To enhance these sensors, researchers are taking advantage of easy preparation of redox-active Salen-type Schiff bases with redox-active metal complexes [6,7]. The nitro groups promote these compounds to have high electron transfer ability [8], and the nanomaterials such as NiO and Bi(III) improve their conductibility and catalytic activity [9–11]. Adding these components influences hybrids which powerfully detect paracetamol and in the presence of other compounds [12–14]. Their surface characteristics can also be enhanced by ion beam irradiation [15,16]. This study focuses on using Bi(III) Schiff base–NiO composites to detect paracetamol under acidic conditions, aiming to build a fast, sensitive, and reliable electrochemical sensor.

## MATERIAL AND METHODS

## Materials

Bi(NO<sub>3</sub>)<sub>3</sub>, NiCl<sub>2</sub> .6H<sub>2</sub>O, NaOH, methanol, ethanol, salicylal aldehyde, nitro salicylaldehyde, ethylene diamine are purchased from e-merk and used as such.

#### Preparation of NiO

5.94 g (0.1M) of NiCl<sub>2</sub> .6H<sub>2</sub>O along with 1 g of NaOH pellet added and makeup in 250 ml of volume then stirring continuously for two hours, Then green gel was formed and washed with water along with ethanol and dried. Then Calcination process was carried out at 450 c in oven, Finally, the green coloured sample changed into black powder and grained with morter which was in nano sized.

#### Preparation of salen;

The salen ligand was prepared by refluxing of a mixture of 3.66 g (3.1ml) of salicylal aldehyde and 0.9g(1.0ml) of ethylene diamine(1:2 ratio) in 150 ml of methanol for one hour.

After completion of reaction the yellow crystalline solid (Salen) was collected and washed with cold methanol and dried in air.

International Journal of Environmental Sciences ISSN: 2229-7359 Vol. 11 No. 16s,2025

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#### Preparation of 5,5' Dinitro salen:

3.04 gm of 5 nitro salicylaldehyde refluxing with 0.9g (1.0ml)of ethylene diamine (1:2 ratio) in 150 ml of methanol for one hour. The pale yellow crystalline solid (Nitro salen) was collected and washed with cold methanol and dried in air.

### Preparation of Bismuth salen complex (SBC):

0.9693g of Bi(NO<sub>3</sub>)<sub>3</sub> in ethanol refluxed with 0.5366g of salen for 2 hours with constant stirring in oil bath and the product was collected by filtration and washed with cold ethanol for 10 minutes and dried in air, then white coloured complex was obtained

## Preparation of 5,5' Nitro salen Bismuth complex (Nitro SBC):

0.9693g of Bi(NO<sub>3</sub>)<sub>3</sub> in ethanol refluxed with 0.4852g of Nitro salen for 2 hours with constant stirring in oil bath and the product was collected by filtration and washed with cold ethanol for 10 minutes and dried in air, then pale yellow coloured complex (Nitro SBC)was obtained.

#### Preparation of NiO -Nitro SBC nano composite:

By using Co-precipitation method, nano sized metal oxide (NiO) dissolved in base medium like NaOH (precipitating medium) was mixed with prepared 5,5'Nitro salen Bismuth Complex(Nitro SBC)in fixed proportion of 1:1 which results black coloured gelly nano composites through Calcination at 120° C along with Centrifugation of 100 rpm, The dried NiO-Nitro SBC was obtained

#### Electrochemical characterization

The electrochemical measurements were carried out by CHI 650C electrochemical work station (CH Instruments, Inc., U.S.A). Electrochemical studies were carried out in a conventional undivided three electrode cell using modified glassy carbon electrode (GCE) as a working electrode (area 0.0341 cm²), Ag|AgCl as a reference electrode and Pt wire as a counter electrode. Prior to each electrochemical experiment, the electrolyte solutions were deoxygenated with pre-purified nitrogen for 10 min unless otherwise specified. Were prepared and used for different pH solution as supporting electrolyte for throughout the electrochemical studies. All the experiments are carried out three times and the average value is used to plot figures and calibration plots.

#### Fabrication of modified electrods

About 2  $\lg$  of compounds suspension in 2 mL DI water. The resultant mixture was stirred at room temperature for 48 h to obtain a putty form of nanocomposite. This nanocomposite was dried overnight at 25 °C in order to evaporate the solvent. The drop-cast method was used for the modification of the electrode. The surface of GCE was gently polished with 0.3  $\mu$ m alumina slurries dispensed on a polishing pad prior to rinsing with distilled water. The GCE-working electrode was then sonicated in ethanol for 5 min and in distilled water again for 5 min to remove any particles that may have been trapped on the surface. Following this, a 20- $\mu$ L aliquot of each of these dispersions was dropped onto the surface of the polished GCE and dried at 50 °C to obtain the modified electrodes used for the analytical studies

#### **RESULTS AND DISCUSSION**

## Cyclic voltammetric studies of Paracetamol on Modified Electrodes

## NiO/GC modified Electrodes

Figure 1 shows the CV profiles of the bare GCE and NiO-modified GCE at the scan rate 25 and 100 mV/s in pH 1.0. The CV curves clearly indicate enhanced redox behavior for NiO/GCE, with a more pronounced oxidation peak and higher anodic current. The oxidation current increased from +0.85  $\mu$ A (GCE) to +1.35  $\mu$ A (GCE/NiO), suggesting more efficient electron transfer. This enhancement is attributed to the NiO nanoparticles creating a porous surface with active defect sites that facilitate charge movement [17–21]. The shift in peak potential and the increased peak height in Figure confirm the improved electrocatalytic activity of the modified electrode.

#### Nitro Salen/GC modified Electrodes:

Figure 2 presents the CV responses for the GCE and Nitro Salen/GC electrodes at both scan rates 25 and 100 mV/s in pH 1.0. The Nitro Salen-modified electrode exhibits sharper and better-defined peaks, with higher oxidation currents. At 100 mV/s, the oxidation peak occurs at 0.48 V with a current of 2.1  $\mu$ A for Nitro Salen/GCE, in contrast to 1.6  $\mu$ A at 0.43 V for GCE. These differences arise from  $\pi$ - $\pi$  stacking interactions between paracetamol and the aromatic Schiff base, hydrogen bonding from

International Journal of Environmental Sciences ISSN: 2229-7359 Vol. 11 No. 16s,2025 https://theaspd.com/index.php

functional groups, and the strong electron-withdrawing nature of the nitro group, all of which accelerate electron transfer. The Bi(III) center within the ligand framework may also enhance catalytic oxidation through intermediate stabilization [20, 22–24]. The distinct redox peaks in Figure 2 validate the superior electrocatalytic behavior of the Nitro Salen system.

#### NiO-Nitro Salen/GC modified Electrodes

Figure 3 displays the CV curves of GCE compared with the NiO-Nitro Salen composite electrode at 25 and 100 mV/s in pH 1.0. The modified electrode demonstrates significantly enhanced peak current and improved redox behavior across both scan rates. At 100 mV/s, the oxidation current increases from  $\pm$ 0.36  $\pm$ 0  $\pm$ 0.58  $\pm$ 4 (NiO-Nitro Salen/GCE). The broadened and more intense peaks in the CV curves suggest greater electron mobility and surface reactivity. This enhanced response is attributed to the combined effects of the NiO nanostructures and the nitro-Schiff base, which together increase the active surface area and provide favorable sites for Paracetamol oxidation [25–27]. The smoother CV transitions and increased peak symmetry in Figure 3 confirm the composite's electrocatalytic efficiency.

#### Nitro SBC/GC modified Electrodes

Figure 4 illustrates the cyclic voltammograms of GCE and Nitro SBC/GCE, At scan rate of 25 and 100 mV/s, the Nitro SBC-modified electrode delivers an anodic peak current of 1.48  $\mu$ A at +0.22 V, almost triple that of the unmodified GCE. The CV profile displays clear peak separation, indicating improved redox kinetics. However, at 100 mV/s, the current response diminishes slightly, likely due to reduced surface conductivity or structural degradation at higher scan rates. The sharp peak observed at 25 mV/s in the modified electrode indicates rapid and favorable redox transitions, making this scan rate optimal for Paracetamol detection using Nitro SBC [28–30]. Figure 4 emphasizes the kinetic advantages of this electrode at lower scan speeds.

#### NiO-NitroSBC/GC modified Electrodes

Figure 5 compares the CV curves of GCE and the NiO-Nitro SBC-modified/GCE. Among all tested materials, this composite shows the highest redox activity. At 100 mV/s, the oxidation peak current rises to  $+0.91~\mu A$  and the reduction peak reaches  $-0.85~\mu A$ , compared to  $+0.74~\mu A$  and  $-0.69~\mu A$  for GCE. The CV curves exhibit well-resolved and symmetrical redox peaks, suggesting enhanced reversibility and faster electron transfer. The superior performance is due to the synergistic interaction between NiO nanoparticles and the nitro-functionalized Schiff base, which enhances both the conductivity and electrocatalytic selectivity of the electrode. Additionally, the nanostructured morphology likely increases the density of electroactive sites [31–33]. The clear enhancement observed in Figure 5 supports the potential of NiO-Nitro SBC as an efficient sensor material.

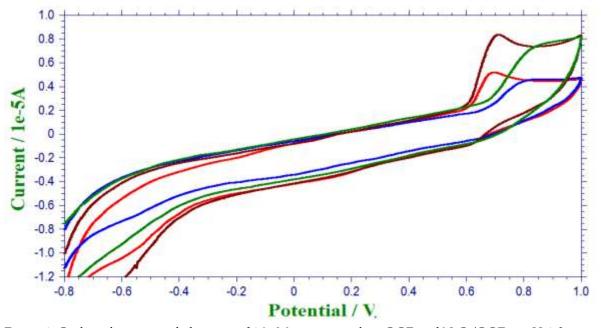


Figure 1. Cyclic voltammetric behaviour of 16  $\mu M$  paracetamol on GCE and NiO/GCE in pH 1.0 at scan rate 25 and 100 mV/s

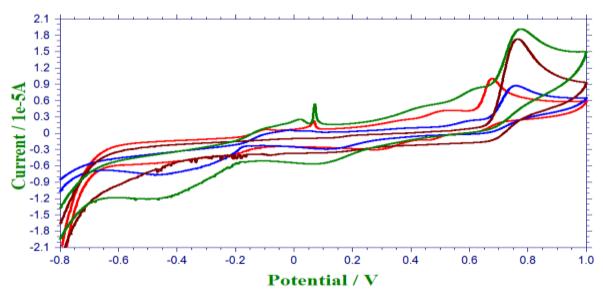


Figure 2. Cyclic voltammetric behaviour of 16  $\mu M$  paracetamol on GCE and Nitro Salen/GC modified Electrodes in pH 1.0 at scan rate 25 and 100 mV/s

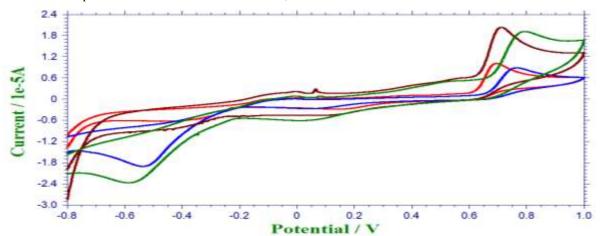


Figure 3. Cyclic voltammetric behaviour of 16  $\mu M$  paracetamol on GCE and NiO-Nitro Salen/GC in pH 1.0 at different scan rate as 25 and 100 mV/s

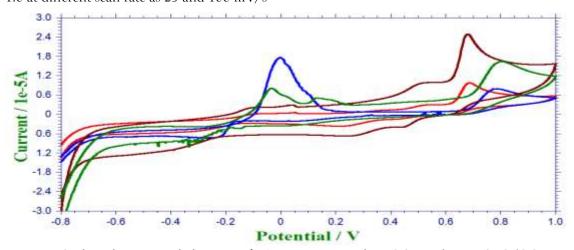


Figure 4. Cyclic voltammetric behaviour of 16  $\mu M$  paracetamol on GCE and Nitro SBC/GCE in pH 1.0 at different scan rate like 25 and 100 mV/s.

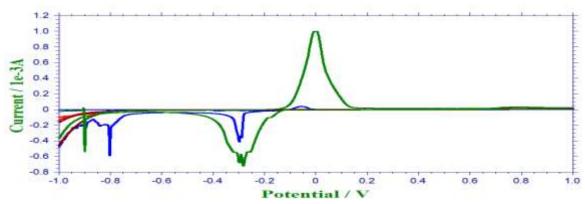


Figure 5. Cyclic voltammetric behaviour of 16  $\mu M$  paracetamol onGCE and NiO-Nitro SBC/GCE at 100 mV/s in pH 1.0

### DPV studies of Paracetamol Sensing on Modified Electrodes

Paracetamol detection at pH 1.0 using DPV showed that modifying GCEs significantly improved performance (Figs. 6). The bare GCE gave a weak signal (3.4  $\mu$ A), while NiO increased it to 6.1  $\mu$ A [34]. Adding a Schiff base raised it to 7.5  $\mu$ A [35], and incorporating a nitro group boosted it further to 9.4  $\mu$ A [36]. Without NiO, the Nitro SBC electrode showed 6.9  $\mu$ A, highlighting the role of nanoparticles. The best result came from NiO–Nitro SBC, reaching 10.2  $\mu$ A at +0.44 V, due to the combined effects of NiO conductivity and Schiff base catalysis [37, 38]. The calibration curve (10–50  $\mu$ M) showed strong linearity (R<sup>2</sup> = 0.974), with a sensitivity of 0.182  $\mu$ A  $\mu$ M<sup>-1</sup> cm<sup>-2</sup> and an LOD of 0.25  $\mu$ M, confirming its potential for trace detection.In, sensor performance followed the order: Bare GCE <NiO-NiO-Salen < Nitro SBC <NiO-Nitro Salen <NiO-Nitro SBC, confirming the advantage of combining functional ligands with nanomaterials for enhanced electrochemical sensing. From the linear plot of ip vs. concentration (Fig. 7) it was seen that the peak current increased with increase in concentration. The straight line plot showed R<sup>2</sup> values of 0.9739. The equations was found to be y = 0.079x +8.81.

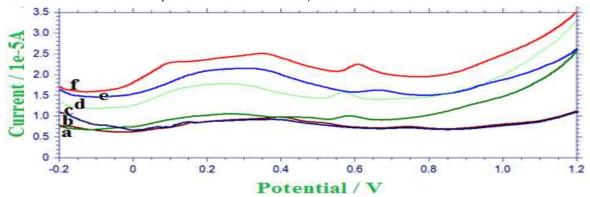


Figure 6. Differential pulse voltammetric behaviour of 10  $\mu$ M paracetamol on a) GCE b) NiO/GCE c) Nitro Salen/GCC d) NiO-Nitro Salen/GC e) Nitro SBC/GCE f) NiO-Nitro SBC/GCE in pH 1.0 under optimum condition.

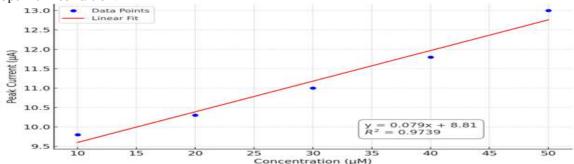


Fig. 7: Calibration curve of NiO-Nitro SBC/GCE, showing the linear relationshIp between current and different concentration

SWV studies of Paracetamol Detection of on Modified Electrodes

International Journal of Environmental Sciences ISSN: 2229-7359 Vol. 11 No. 16s,2025 https://theaspd.com/index.php

At pH 1.0, SWV revealed that modifying GCEs with Nitro-Salen, NiO, and their composites significantly boosted p-aminophenol detection (Figs. 8). Nitro-Salen raised the current from 60 to 90  $\mu$ A, and Nitro-NiO reached 85  $\mu$ A. The best response came from Nitro-SBC (100  $\mu$ A), showing strong electrocatalytic activity. The NiO-Nitro SBC electrode doubled the current vs. bare GCE in an extended range (5.5 vs. 2.5  $\mu$ A). The NiO-5,5'-dinitro SalenBi(III) sensor showed excellent linearity (R<sup>2</sup> = 0.9910), sensitivity (0.55  $\mu$ A/unit), and a low LOD of 0.082 units (Fig. 8). These findings confirm that combining NiO and nitro-Schiff bases improves redox kinetics and sensing in acidic media [20, 39-42].

A plot of peak current versus concentration was drawn (Fig. 9). The linear dependence of peak current with concentration was understood from the straight line with good correlation. The R-squared values corresponding to obtained to be 0.999 respectively.

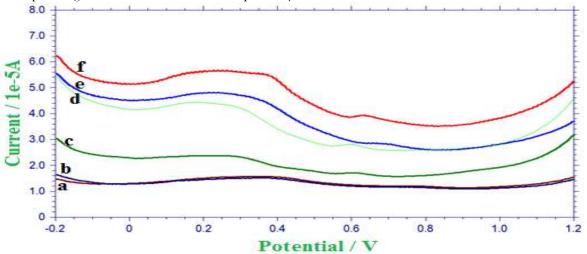


Figure 8. Square wave voltammetric behaviour of  $10\,\mu M$  paracetamol on a) GCE b) NiO/GCE c) Nitro Salen/GCC d) NiO-Nitro Salen/GC e) Nitro SBC/GCE f) NiO-Nitro SBC/GCE in pH 1.0 under optimum condition

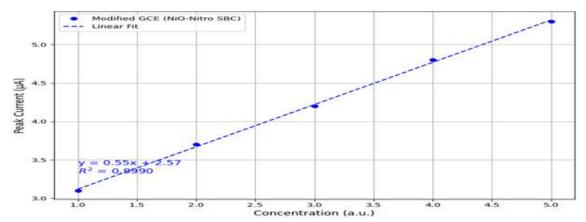


Fig. 9: Calibration curve of NiO-Nitro SBC/GCE, showing the linear relationshIp between current and different concentration

Table 1: Electrochemical Performance of NiO-Nitro SBC Electrode for Paracetamol Detection

Technique	16 µM Con	16 μM Conc				
CV	Peak	Current:	~0.91	μΑ		
	Peak	Potential:	~+0.48	V		
	Irradiation:	Irradiation: 100 MeV				
DPV	Peak	Current:	~1.48	μΑ		
	Peak	Potential:	+0.22	V		
	Sensitivity:	0.182	$\mu A/\mu M/cm^2$			

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ISSN: 2229-7359 Vol. 11 No. 16s,2025

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	LOD:	0.25		μΜ
	R <sup>2</sup> : 0.974			
SWV	Peak	Current:	~5.5	μΑ
	Peak	Potential:	~+0.44	V
	LOD:	0.082		$\mu M$
	Sensitivity:	0.55		$\mu A/\mu M$
	R <sup>2</sup> : 0.9910			

#### CONCLUSION

The NiO-Nitro Schiff base Bi(III) complex (NiO-Nitro SBC) demonstrated excellent electrochemical performance for paracetamol detection across CV, DPV, and SWV techniques. In DPV at 77  $\mu$ M, it delivered the highest peak current (67.8  $\mu$ A at 0.59 V), with strong sensitivity (0.052  $\mu$ A/ $\mu$ M), low LOD (0.29  $\mu$ M), and outstanding linearity (R² = 0.9993). At 16  $\mu$ M, the sensor remained highly responsive, showing a sensitivity of 0.182  $\mu$ A/ $\mu$ M/cm² and an LOD of 0.25  $\mu$ M. SWV at this lower concentration yielded the lowest LOD (0.082  $\mu$ M), while the 77  $\mu$ M test still produced a strong signal (57.6  $\mu$ A, R² = 0.9998). Overall, the NiO-Nitro SBC-modified GCE proved to be a robust and versatile platform, with DPV at 77  $\mu$ M offering the best combination of sensitivity, detection limit, and linearity ideal for pharmaceutical sensing applications.

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