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# Highly Sensitive Endocrine-Disrupting Chemicals Pollutants Detection on ZnO Enhanced Nano Carbon Electrode

# La Ode Agus Salim,<sup>1,\*</sup> Kurnia Sri Yunita,<sup>1</sup> and Muhammad Nurdin<sup>2,3</sup>

<sup>1</sup>Department of Chemistry, Faculty of Science Technology and Health, Institut Sains Teknologi dan Kesehatan (ISTEK) 'Aisyiyah Kendari, Kendari, 93116 – Southeast Sulawesi, Indonesia <sup>2</sup>Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Halu Oleo, Kendari 93231 – Southeast Sulawesi, Indonesia <sup>3</sup>Nickel Research Institute, Universitas Muhammadiyah Kendari, Kendari 93127-Southeast Sulawesi, Indonesia

\*Corresponding Author, Tel.: +62821-9435-2553 E-Mail: <u>agus.123742@gmail.com</u>

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Abstract- An innovative investigation was conducted to characterize a Zinc Oxide-modified nano carbon (NC-ZnO) electrode for the development of an initial model for detecting Endocrine-Disrupting Chemicals (EDCs). NC-ZnO synthesis was carried out by the hydrothermal method and characterized. The particle size of the carbon-modified ZnO is determined to be 22.85 nm. The electrochemical performance evaluation demonstrates that the ZnO modification significantly improves electron transfer and enhances the electrochemical performance of NC electrodes in terms of increased peak currents and narrower current peaks. The results of our study indicate that the NC@ZnO composite electrode is effective in detecting fipronil, with efficient electron transfer occurring at a low oxidation potential. Additionally, the electrode demonstrates a highly linear sensitivity to fipronil concentration in the range of 10 to  $10^{-3} \mu g/L$ , and it boasts an impressive limit of detection (LoD) of 0.00393  $\mu g/L$ . Moreover, the NC@ZnO composite electrode shows exceptional stability in detecting fipronil, as evidenced by the low stability coefficients of %RSDR (0.048) and %PRSDR (0.13). These findings suggest that the newly developed NC@ZnO composite electrode holds great promise as a sensitive tool for detecting fipronil in the environment, thus potentially helping to reduce its negative impacts on human health and ecosystems.

Keywords- NC@ZnO; Composite; Carbon; Fipronil; Sensor

# **1. INTRODUCTION**

Fipronil, a synthetic insecticide widely used in agriculture and veterinary applications, is categorized as an endocrine-disrupting chemicals (EDCs) and poses significant risks to both human health and the environment [1]. Its potential to interfere with the endocrine system raises concerns about adverse health effects in humans and wildlife, including hormone imbalances, reproductive disorders, and developmental abnormalities [2]. Regulatory authorities establish a threshold limit value (TLV) for fipronil, representing the maximum safe concentration for human exposure over a specified period, considering its bioaccumulative nature and potential endocrine-disrupting effects [3]. Proper control and monitoring of fipronil levels are essential to mitigate its impact on ecosystems and human health, ensuring responsible use in the environment and safeguarding the well-being of both humans and wildlife.

The detection of fipronil is commonly carried out using chromatographic techniques such as thin-layer chromatography (TLC) [4], high-performance liquid chromatography (HPLC) [5], and gas chromatography-mass spectrometry (GC-MS) [6]. However, these chromatographic methods are complex, involve time-consuming sample preparation, and are vulnerable to measurement errors [7]. A practical alternative that has been explored is electrochemical methods, such as voltammetry and amperometry, which offer rapid and sensitive detection of fipronil with simpler sample preparation requirements [8-11]. Additionally, electrochemical processes are cost-effective and require relatively inexpensive equipment, making them more accessible for routine analysis in various environmental and food safety applications [12,13]. Continuous efforts to develop and validate alternative electrochemical methods are still necessary to ensure reliable and efficient fipronil detection.

A promising and emerging alternative technique for detecting fipronil involves voltammetry using a nanocarbon working electrode that is modified with Zinc Oxide (NC@ZnO). ZnO is known for its excellent catalytic properties, which synergistically enhance the electrocatalytic activity of the modified electrode [14]. The hydrothermal carbon modification process includes controlled synthesis of carbon nanomaterials and ZnO under specific temperature conditions, resulting in a highly porous and conductive nanostructured surface [15]. This modification enhances the electrode's electrocatalytic activity, creating a larger active area for fipronil adsorption and detection, leading to increased sensitivity and stability of the voltammetric detection system.

In our study, we present highly sensitive fipronil detection using NC@ZnO as the working electrode. We observed that the presence of ZnO is crucial for sensing sensitivity, which increases with higher ZnO concentrations, peaking at 0.00393  $\mu$ g/L. At this concentration, fipronil can be easily detected in the supporting electrolyte, demonstrated by a high anodic current at the oxidation potential of fipronil over this modified electrode. The investigation revealed commendable stability coefficients for both the relative standard deviation of reproducibility (RSDR) and the randomized strategic demand reduction (PRSDR), yielding

values of 0.048% and 0.13%, respectively. This innovative approach shows promise for rapid and accurate fipronil detection in environmental and agricultural samples. Additionally, the cost-effectiveness and scalability of the hydrothermal carbon modification process make it a practical and attractive choice for widespread application in fipronil monitoring and environmental safety efforts.

# 2. EXPERIMENTAL METHOD

### 2.1. Green synthesis of nano carbon from palm shell

Palm shell samples originating from palm plantations in South Konawe, Southeast Sulawesi, were processed using the Microwave method for NC synthesis. The NC synthesis method was modified by referring to previous research [16,17]. The palm oil shells were cleaned with water to remove dirt and residue. Subsequently, the shells were dried using an oven at a temperature of 70°C and then crushed into powder form. The powder was then carbonized in a furnace at a temperature of around 500°C for 3 hours. Next, the material underwent activation using a microwave (450 Watts for 5 minutes). The NC material was further crushed into smaller sizes to produce nanoparticles. The NC with graphite was mixed with water, and the solution was separated using centrifugation or filtration to remove any impurities. After washing, the NC with graphite was dried using an oven and then stored in an airtight container to prevent moisture and contamination.

#### 2.2. Synthesis of of NC@ZnO electrode composite

NC and ZnO powders were separately prepared in 200 mesh particle size. This preparation method was modified from previous studies [18]. The preparation of the composite with different NC:ZnO ratios (3:2). Subsequently, these powders were mixed with paraffin oil having a density of 0.88 g cm<sup>-3</sup> in a glass beaker. To ensure the formation of a homogeneous composite, the mixture was stirred using a hot plate stirrer at a temperature of 80°C and a stirring speed of 100 rpm for 30 minutes. The resulting composite was then packed into a glass tube with a diameter of approximately 3 mm, which served as the electrode body. To achieve a smooth surface, the composite was polished mechanically and refined using sandpaper, followed by connection to a copper wire for electrical contact. All chemical reagents utilized in this process were acquired from Aldrich (USA) and used without any further purification. The experimental design of NC@ZnO electrode preparation and electrochemical system showed in Figure 1.

## 2.3. Characterization of composite

The morphology and composition of the NC@ZnO composites was analyzed using Scanning Electron Microscopy- Energy Despersive X-ray (SEM-EDX) on a HITACHI SU3500. The chemical structure of the composite was evaluated via Fourier Transform Infrared spectroscopy (FTIR) using a Shimadzu IR Affinity-1S system. the characteristics of phase and crystallinity were analyzed using X-ray diffraction spectroscopy (XRD) Philips which utilized CuK $\alpha$  irradiation with a wavelength of 0.154 nm and the scanning rate utilized was as slow as 2° per minute. The electrochemical properties of the composite were investigated using cyclic voltammetry technique with a potentiostat DY2100. The probe used for the electrochemical analysis was a glass container with a diameter of approximately 2.00 cm and a height of around 3.50 cm. The top cover of the probe had three holes to accommodate the electrodes, including both working and auxiliary electrodes, as well as a reference electrode.

# 2.4. Electrochemical performance of NC@ZnO for fipronil detection

The detection of fipronil was conducted utilizing a cyclic voltammetry technique with a 3electrode setup. The NC@ZnO composite electrode was designated as the working electrode, and 0.1 M NaNO<sub>3</sub> was utilized as the supporting electrolyte. The electrochemical measurements were performed under a potential window of  $\pm 0.8$  V using an instrument with a scan rate of 0.02 V for the fipronil detection procedure. To ensure the reliability and consistency of the electrochemical results, 20 repetitions of the experiment were performed.



**Figure 1.** Experimental design of NC@ZnO electrode preparation and  $Fe(CN)_6^{3-}/Fe(CN)_6^{4-}$  electrochemical system

# **3. RESULTS AND DISCUSSION**

# 3.1. Characterization of chemical bond and morphology electrode

The SEM analysis results are presented for two categories of composites: one incorporating NC (Figure 2a) and the other combining NC with ZnO (Figure 2b). NC is distinguished by its compact carbon particle structure of uniform size. Additionally, the morphology exhibits a distinct NC layer unique and consistent with prior studies [19]. Conversely, ZnO nanoparticles display surfaces with pores formed by particles of varying sizes on the surface of NC. This porous structure encourages interactions between the analyte and electrode surfaces, thereby facilitating enhanced movement of analyte molecules and electron transfer on the electrode surface [20,21]. The incorporation of NC and ZnO nanoparticles minimally alters the morphology. ZnO nanoparticles disperse uniformly onto the NC particles, attributed to the smaller size of the ZnO nanoparticles. This attribute contributes to achieving a homogenous composite morphology of NC and ZnO, highly desirable for electrode modification. Such uniformity significantly enhances electrode conductivity, a critical aspect of improving electrode performance.



**Figure 2.** Surface morphology of (a) NC, (b) NC@ZnO, c and d are EDX mapping particle NC@ZnO composite

Furthermore, an extended discussion can be provided, highlighting the relevance of the observed composition for the potential application of the composite electrode in electrochemical sensor devices. The conducted EDX analysis assumes paramount significance in confirming the successful integration of Zn into the intricately structured carbon matrix, as visually elucidated in Figure 2c. This analytical approach has become indispensable in the domain of materials science due to its capability to identify and quantitatively assess elemental compositions within a given specimen. The EDX spectrum depicted in Figure 2d imparts comprehensive insights into the elemental components present within the nanostructure, encompassing Carbon (C), Oxygen (O), and Zinc (Zn). The relative proportions of these constituents are quantified at 87.27%, 6.22%, and 3.69%, correspondingly. These distinctive ratios serve as strong evidence supporting the successful synthesis of the NC@ZnO composite electrode [22]. From an application perspective, the achieved composition holds significant promise for utilization in electrochemical sensor devices. The presence of Zn within the composite structure can augment the electrode's electrocatalytic properties due to the intrinsic catalytic activity of ZnO. This can be particularly advantageous in electrochemical sensors, where efficient electron transfer at the electrode interface is essential for rapid and accurate sensing [23,24]. Moreover, the NC component in the composite is known for its excellent electrical conductivity and large surface area, which further contributes to enhanced sensor performance [25].



Figure 3. FTIR spectra of NC and NC@ZnO

To confirm the chemical bonding within the composite electrode, an FTIR analysis was conducted. Figure 3 showed the FTIR spectra of NC and NC@ZnO composites. Based on the FTIR characterization results, peaks were observed at wavenumbers of 3384 cm<sup>-1</sup>, 1799 cm<sup>-1</sup>, 1426 cm<sup>-1</sup>, 1046 cm<sup>-1</sup>, and 875 cm<sup>-1</sup> in the modified NC@ZnO composite. These peaks indicate

potential bonds formed within the material. The peak at 3384 cm<sup>-1</sup> suggests the presence of hydrogen bonding, while the peak at 1799 cm<sup>-1</sup> indicates the presence of carbonyl groups. The peak at 1426 cm<sup>-1</sup> reveals the existence of double bonds (alkenes) in the carbon structure. The peak at 1046 cm<sup>-1</sup> suggests the presence of ethanol bonds, and the peak at 875 cm<sup>-1</sup> indicates the vibration of methyl groups in the NC. These potential bonds signify structural modifications and functional interactions between ZnO and NC, which can impact surface reactivity, conductivity, and potential applications of the material [26].

The XRD analysis of NC@ZnO characterizes the material's structural properties and size particle of nano. Figure 4 showed the acquired XRD pattern, displaying distinct diffraction peaks associated with ZnO. These peaks, observed at 20 values of 25.196°, 42.747°, 46.627°, and 48.110°, align with the characteristic diffraction pattern of ZnO (refer to Table 1). Consequently, these observations affirm the successful synthesis of carbon-modified ZnO. Notably, the presence of ZnO within the carbon-modified sample is confirmed. The obtained XRD pattern in this study closely resembles patterns documented by earlier researchers, thereby further substantiating the reproducibility of the carbon modification process [27]. Moreover, the particle size of the carbon-modified ZnO sample was determined using the Scherrer equation. By assessing the broadening of the XRD peaks, the particle size was computed to be 22.85 nm. This nanoscale particle size attests to the effective facilitation of ZnO nanoparticle formation through carbon modification. The reduced particle size achieved via carbon modification brings forth various advantages, including heightened surface area and reactivity [28]. These properties hold substantial appeal for diverse applications, encompassing photocatalysis, sensing, and energy storage [29].



Figure 4. XRD spectrum of NC and NC@ZnO

Pos [°2theta]	$\theta$ (radian)	d-Spacing (Å)	Particle size (nm)
25.196°	0.438928	3.4347	34.347
42.747°	0.745115	2.0297	20.297
46.627°	0.813604	1.8644	18.644
48.110°	0.839070	1.8146	18.146

Table 1. Data comparison of °2Theta and d-spacing from NC@ZnO

# 3.2. Fe(CN)<sub>3</sub><sup>-6</sup>/Fe(CN)<sub>4</sub><sup>-6</sup> electrochemical system

Two working electrodes, namely unmodified NC and NC@ZnO were tested using CV in the Fe(CN) $_{6}^{3-}$ /Fe(CN) $_{6}^{4-}$  solution system. The NC electrode was used as a reference to study the effect of ZnO modification, employing the Fe(CN) $_{6}^{3-}$ /Fe(CN) $_{6}^{4-}$  solution due to its solution stability and ease of monitoring the transferred electron count during redox reactions (Figure 5a). The voltammogram of the NC electrode exhibited broad peaks with low redox currents, indicating sluggish electron transfer. This observation was further supported by the oxidation potential (Ea) of 0.53 V and reduction potential (Ec) of -0.35 V. The broadening of the voltammogram peaks led to inaccuracies in determining the current values for oxidation and reduction processes. However, the electrochemical performance of NC was improved through modification with ZnO. NC@ZnO facilitated accelerated electron transfer and increased peak currents for both oxidation and reduction reactions. The addition of ZnO also resulted in narrower current peaks. The enhanced electron transfer rate can be attributed to the high conductivity of ZnO and increased surface to volume ratio of the NC electrode [22]. With the inclusion of ZnO, electron transfer became more efficient, leading to an increase in peak currents.



**Figure 5.** Cyclic voltammogram of 0.1 M  $K_3$ [Fe(CN)<sub>6</sub>] solution on composites, (a) NC to anatase ZnO ratio of 3:2 electrode and composite sample with a ratio of 3:3 samples for comparison. The scan rate is 0.1 V/s. b) different scan rate for NC to ZnO ratio of 3:2 electrode

The comparison between the anodic peak current ( $I_{pa}$ ) and cathodic peak current ( $I_{pc}$ ) indicated that the reaction occurring in the Fe(CN)<sub>6</sub><sup>3-</sup>/Fe(CN)<sub>6</sub><sup>4-</sup> system at the Gr electrode was irreversible, as evidenced by  $I_{pa}/I_{pc} \neq 1$ . This suggests that the reaction does not easily proceed in the forward or reverse direction spontaneously [30]. The influence of varying scan rates on the behavior of the NC@ZnO electrode immersed in a 0.1 M K<sub>3</sub>[Fe(CN)<sub>6</sub>] solution is depicted graphically in Figure 5b. The most prominent peaks, corresponding to oxidation and reduction processes, were identified using a scan rate of 0.02 V/s. However, it is worth noting that elevated scan rates can impact electrochemical performance due to the rapid migration of ions toward the electrode interface. This phenomenon subsequently reduces the rate of ion transport from the electrolyte to the electrode, as evidenced by a scan rate of 0.1 V/s [31,32]. Consequently, a scan rate of 0.02 V/s was deliberately selected to assess the sensor's efficacy in detecting fipronil. This specific scan rate was found to optimize the charging and discharging capabilities of the working electrode, indicating its rapid response.

# **3.3. Determination of fipronil**

In the final phase of this study, a comprehensive assessment was conducted to evaluate the responsiveness of the NC@ZnO electrode towards varying concentrations of fipronil spanning a range from 10 to  $10^{-3} \mu g/L$ . By utilizing a scan rate of 0.02 V/s, a voltammogram capturing the oxidation process of fipronil at different concentrations was obtained, as illustrated in Figure 6a. The graphical representation depicting the correlation between the anodic current and fipronil concentration is presented in Figure 6b. Remarkably, the results establish a robust linear relationship between the anodic current and fipronil concentration within the concentration range of 10 to  $10^{-3} \mu g/L$ . The limit of detection (LoD) for the fipronil compound was determined to be an impressively low value of 0.00393  $\mu g/L$ .

Working electrode	Method	LOD (µg/L)	References
Multi-walled carbon-nanotube (MWCNT)-modified glassy carbon electrodes (GCEs)	Amperometric	1.88	[10]
Graphite-polyurethane (GPU)	CV and SWV	0.80	[33]
ZnO/g@C <sub>3</sub> N <sub>4</sub> / glassy carbon electrode	Electrochemiluminscence (ECL)	0.65	[34]
Ilmenite-CPE	CV	1.04	[35]
Al(III)-TiO <sub>2</sub> /Graphene	CV	0.0164	[36]
CN@ZnO	CV	0.00393	This Work

 Table 2. Comparative analysis of fipronil detection outcomes across diverse working

 electrodes

This significant achievement not only surpasses previously reported findings but also remains comfortably below the established permissible threshold for fipronil compounds within the environment (Table 2).

Furthermore, a thorough exploration into the stability of the NC@ZnO electrode for fipronil detection was systematically conducted. The investigation revealed commendable stability coefficients for both the relative standard deviation of reproducibility (RSDR) and the randomized strategic demand reduction (PRSDR), yielding values of 0.048% and 0.13%, respectively, as visually demonstrated in Figure 6c. In the realm of electrochemical detection, the susceptibility of active sites to potential degradation, such as susceptibility to poisoning from adsorbed redox species, emerges as a pertinent concern [37]. However, the marginal reduction in performance observed over 20 consecutive measurements strongly indicates the high practical suitability of this novel electrode. This observation underscores the remarkable resistance of the NC@ZnO electrode to potential degradation, positioning it as a promising candidate for practical applications.



**Figure 6.** (A) CV curves of fipronil at different concentrations using the NC@ZnO electrode, (B) the related graph depicting the relationship between peak current (I<sub>pa</sub>) and fipronil concentration, and (C) a histogram presenting the reproducibility measurements

In light of the collective findings, it can be inferred that the NC@ZnO electrode embodies an impressive fusion of sensitivity and stability. This distinctive combination firmly situates the electrode as a highly auspicious contender for the detection of fipronil compounds within the environmental milieu.

#### 4. CONCLUSION

In this study, an innovative investigation was conducted to characterize an NC@ZnO electrode, aimed at establishing an initial model for detecting EDCs. The synthesis of NC@ZnO was achieved through the hydrothermal method, followed by thorough characterization using techniques such as SEM-EDX, FTIR, and XRD. The determined particle size of the carbon-modified ZnO was found to be 22.85 nm. Through extensive electrochemical performance evaluation, it was observed that ZnO modification imparted notable enhancements in electron transfer efficiency, thereby elevating the electrochemical capabilities of NC electrodes. This enhancement was evidenced by increased peak currents and narrower current peaks. The experimental outcomes highlight that the NC@ZnO composite electrode exhibits effective performance in the detection of fipronil, particularly with proficient electron transfer occurring at a low oxidation potential. Furthermore, the composite electrode exhibited a remarkable linear sensitivity to fipronil concentration within the range of 10 to  $10^{-3} \mu g/L$ . Notably, the electrode demonstrated an impressive limit of detection (LoD) of 0.00393 µg/L. Noteworthy stability was also observed in the detection of fipronil compounds, as evidenced by the low stability coefficients of %RSDR (0.048) and %PRSDR (0.13). These significant findings underscore the potential of the newly developed NC@ZnO composite electrode as a highly sensitive tool for detecting fipronil in the environment. This advancement holds promise in mitigating the adverse impacts of fipronil on human health and ecosystems.

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## **Declarations of interest**

The authors declare that we have no competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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