A Sensor for the Determination of Lindane Using PANI/Zn, Fe(III) Oxides and Nylon 6,6/MWCNT/Zn, Fe(III) Oxides Nanofibers Modified Glassy Carbon Electrode

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A simple reproducible and environmentally friendly PANI/Zn, Fe(III) and Nylon 6,6/MWCNT/Zn, Fe(III) oxides nanofibers modified glassy carbon electrode was prepared and used for the electrochemical reduction of lindane. The modified electrodes offer a high sensing current for lindane. The modified electrodes were highly stable with respect to time, so that the single electrode can be used for the multiple analysis of the lindane sample. Cyclic voltammetry and square wave voltammetry were used as the sensing techniques. The dynamic range for the lindane determination was between $9.9 \times 10^{-12}$ mol/L and $5 \times 10^{-6}$ mol/L with detection limits of 51 and 32 nM for Nylon 6,6/MWCNT/ZnO and Nylon 6,6/MWCNT/Fe$_3$O$_4$ sensors, respectively. The LoD value reveals that the best electrode is Nylon 6,6/MWCNT/Fe$_3$O$_4$. The analytical utility of the proposed method was checked with drinking water samples.

1. Introduction

Organochloride insecticides, such as hexachlorocyclohexane generally known as lindane, are serious environmental pollutants and are normally extremely resistant to biodegradation [1]. Lindane (γ-hexachlorocyclohexane) is also used in formulation of lotions and shampoos to treat lice and scabies [2]. Lindane has been known to cause immuno-toxicity and retards reproduction and growth of animals, aquatic organisms, and human [3]. Large scale production and application of this insecticide in agriculture deteriorate the environment owing to its long period of persistence [4] and the fact that there is no natural degradation of lindane. Moreover, it has moderate volatility and can be transported by air to remote locations [5]. It has deteriorating effects on the central nervous system of mammals, provoking seizures and in some cases causing death [6]. Microbial degradation of γ-HCH under aerobic conditions has been reported [7]. Anaerobic degradation of lindane with 95% removal efficiency has also been reported [8]. Different methods have been used for the detection and estimation of lindane such as colorimetric measurement of chloride ions [9, 10], phenol red based colorimetric method [11], colorimetric measurement using zinc in acetic acid [12], determination of complete mineralization to 14CO$_2$ [13], thin-layer chromatography [9, 10], and gas chromatography [13, 14]. The major demerits of these methods have to do with the use of rigorous approach involving solvent extraction, which is time-consuming, very expensive, and also not suitable for on-site monitoring [14]. Therefore, quantitative analysis of lindane is an important area in the context of environmental protection.

Nanocomposites of different functionalities have been widely explored for electrochemical detection of biological...
The application of nanofibers in these areas is also reported [17]. Nanofibers are exciting materials used for several value-added applications [18, 19]. Nanofibers also possess unique characteristics such as high surface area per unit mass, high porosity, excellent structural mechanical properties, high axial strength combined with extreme flexibility, and the ease of surface chemistry modification in order to accommodate various functionalities such as metal and metal oxides nanoparticles. Electrospinning has been an effective method for the fabrication of fibres of nanosize diameter [20–22]. The use of multiwalled carbon nanotube in functionalization of materials used as biological and environmental sensors cannot be overemphasized due to the attractive features of the multiwalled carbon nanotubes (MWCNTs) including their unique mechanical and electrical properties [23]. Nanocomposite electrodes made of carbon nanofibers and paraffin wax were characterized and investigated as novel substrates for metal deposition and stripping processes. The electrochemical properties of CNF-ceramic composite electrodes [24, 25] and CNF-polystyrene composite electrodes [26] have been reported. Maldonado and Stevenson directly prepared carbon nanofibers electrodes by pyrolysis of iron(II) phthalocyanine on nickel substrates and investigated the electrochemical behaviour and stability of CNF electrodes as oxygen reduction catalysts [27, 28].

Electroanalytical techniques have emerged as a viable alternative to sense this compound quantitatively over other sophisticated analytical techniques such as gas chromatography, liquid chromatography, or gas chromatography-mass spectrometry (GC-MS) due to various advantages like low cost, compact nature, quick response time, low detection limits, selectivity, and high sensitivity [29, 30]. Currently, some researches have examined electrochemical techniques in the detection of chlorobenzene [24, 25].

Therefore, the objectives of this work are to develop a stable and reproducible modified PANI/Zn, Fe(III) and Nylon 6,6/MWCNT/Zn, Fe(III) oxides nanofibers-based sensors for the electrochemical sensing of lindane in aqueous solutions. A comparative study of the electrochemical properties of these modified electrodes was also investigated based on the fact that nanofibers have large surface area and also ease of surface functionalization which can be used to improve the electrochemical properties of bare electrode. To the best of our knowledge, this is the first study on comparative electrochemical behaviour of nanofibers-based sensors functionalized with metal oxides nanoparticles (FeO4, ZnO) towards electrocatalysis of lindane.

2. Experimental

2.1. Materials and Equipment. Lindane, tetrabutylammonium bromide (TBAB), Nylon-6 polymer (average molecular weight of 13,000–15,000), formic acid (HFIP, 99%), aniline (C6H5NH2), ammonium persulfate ((NH4)2S2O8, APS), and hydrochloric acid (HCl) were purchased from Sigma-Aldrich. The pristine multiwalled carbon nanotubes (MWCNTs) were obtained commercially from Aldrich chemicals. A working glassy carbon electrode (GCE, 3 mm diameter), Ag/AgCl, satd KCl reference electrode, and a platinum disk counter electrode (99.999%) were purchased from CH Instrument Inc., US. The salts of the metals, iron(III) chloride FeCl3 and zinc nitrate hexahydrate Zn(NO3)2·6H2O, were obtained from Sigma-Aldrich. Other reagents were also obtained from Sigma-Aldrich and Merck chemicals, respectively. Ultrapure water of resistivity of 18.2 MΩcm was obtained from a Milli-Q Water System (Millipore Corp., Bedford, MA, USA) and was used throughout for the preparation of solutions. All solutions were prepared using double distilled deionized water and purged with pure nitrogen to eliminate oxygen and any form of oxidation during experiment. All other reagents were of analytical grades and were used directly as received from the suppliers without further purification. All electrochemical experiments were performed with nitrogen.

All electrochemical measurements were carried out using AUTOLAB Potentiostat PGSTAT 302 (Eco Chemie, Utrecht, Netherlands) driven by the GPE software version 4.9 in an electrochemical workstation consisting of three-electrode system, a glassy carbon electrode (GCE) as the working electrode, a silver-silver chloride electrode (SCE) as the reference electrode, and a platinum (Pt) wire as the counter electrode. All solutions were deaerated by bubbling nitrogen prior to each electrochemical experiment. Experiments were performed at 25 ± 1°C.

UV-vis absorption spectra were recorded by a UV-1901 UV-vis spectrophotometer (Agilent Technology, Cary series UV-vis spectrometer) using quartz cell with the path length of 1.0 cm. FTIR experiments were performed using Fourier transformed infrared spectrophotometer (Agilent Technology, Cary 600 series FTIR spectrometer, USA). Scanning electron microscope (SEM, JEOL-JSM-6700F), respectively, was used to characterize the chemical change and morphology of the synthesized nanomaterials.

2.2. Methods

2.2.1. Synthesis of Zn and Fe(III) Oxide Nanoparticle. The production unit of ZnO nanostructures consists basically of a jacketed three-neck glass flask and of a magnetic stirrer with temperature control. In the three-neck glass flask, NaOH was dissolved in deionized water to a concentration of 1.0 M and the resulting solution was heated, under constant stirring, to the temperature of 70°C. Another solution of 0.5 M Zn(NO3)2·6H2O was added slowly (dripped for 60 minutes) into the three-neck glass flask containing NaOH aqueous solution under continual stirring. In this procedure the reaction temperature was constantly maintained at 70°C. The suspension formed with the dropping of 0.5 M Zn(NO3)2·6H2O solution to the alkaline aqueous solution was kept stirred for two hours in the temperature of 70°C. The material formed was filtered and washed several times with deionized water. The washed sample was dried at 65°C in oven for several hours [32].

30 mL of 2 mol dm−3 FeCl3 stock solutions, 20 mL of 1 mol dm−3 Na2SO3 stock solution, and 50.8 mL of concentrated ammonia diluted to a total volume of 800 mL were used. Just after the mixing of Na2SO3 and NH3 solution, the color of the solution in the smaller beaker could be seen to alter from light yellow to red, indicating formation of complex
ions. This solution was poured quickly into the diluted ammonia solution under vigorous stirring when the color changed from red to yellow again. A black precipitate formed. Stirring was continued for 30 min. After the reaction, the beaker containing the suspension was placed on a permanent magnet. Black powders could be seen to quickly settle on the bottom of the beaker. The supernatant was discarded and fresh water was added to the beaker; this procedure was repeated several times until most of the ions in the suspension were removed. Dry powders were obtained by filtering and drying at room temperature [33].

2.2.2. Preparation of Polyaniline. Polyaniline nanofibers (PANI) were synthesized according to the procedure reported in literature [34]. 0.298 g aniline was added to 10 mL of 1.0 M HCl aqueous solution. 0.186 g APS was dissolved in 10 mL of 1.0 M HCl aqueous solution. The initiator solution (APS in 1.0 M HCl) was added into the monomer solution (aniline in 1.0 M HCl) all at once. The initial ratio of APS/aniline was 1:4. The polymerization reaction was carried out under static conditions for 1 h at room temperature. The resulting polyaniline precipitate was filtered, washed with deionized water several times, and dried at vacuum condition (50 °C) for 24 h.

2.3. Fabrication of Nylon 6,6/MWCNT/Fe3O4, ZnO and PANI/Fe3O4, ZnO Nanocomposites. 10 wt.% Nylon 6,6 was dissolved in the formic acid at room temperature, and clear solution was obtained by stirring for 3 h. 2.5 mg of metal oxides Fe3O4 and ZnO nanoparticles was added, respectively, into the 10% (w/v) Nylon 6,6 in formic acid. The resulting Nylon 6,6/Fe3O4 solutions were stirred for 5 h and sonicated for 1 h at the ambient conditions to obtain homogeneous solutions. Each resulting solution was loaded to a syringe fitted with a metallic needle. The electrospinning set-up consisted of high voltage supply and an aluminum collecting plate. The flow rate of the polymer solution was controlled using a programmable syringe pump (Model NE-1010, New Era Pump Systems Inc., USA). The solution was electrospun at a positive voltage of 20 kV, the tip-to-collector distance was 10 cm, and the flow rate was 0.2 mL/h. All procedures were carried out at room temperature. The schematic diagram of the electrospinning set-up used in this work is shown in Figure 1 [31]. The nanofibers of Nylon 6,6/Fe3O4 and Nylon 6,6/ZnO are, respectively, doped in MWCNT and sonicated to yield Nylon 6,6/MWCNT/Fe3O4 and Nylon 6,6/MWCNT/ZnO nanocomposites.

Slurry of PANI/MO nanocomposites was prepared by, respectively, mixing 2 mg PANI, 2.5 mg Fe3O4, and ZnO in 1 mL DMF and sonicated for several hours. The resulting mixture is PANI/Fe3O4 and PANI/ZnO nanocomposites.

2.4. Preparation of Modified GC Electrode. The GC electrode was polished with 0.3 μm and 0.05 μm alumina slurries for 3 min each step, followed by thoroughly rinsing with water and sonication in turn with distilled water, ethanol and distilled water for 3 min each before modification. The voltammograms were performed between −2.0 V and 0 V at 25 mV/s. The polished electrode was electrochemically pretreated by cycling the potential scan between −2.0 V and 0 V in 1 mM lindane solution at the scan rate of 25 mV/s to 300 mV/s at bare GCE, GCE/Nylon 6,6/MWCNT/MO, and GCE/PANI/MO, where MO is Fe3O4 and ZnO which were prepared by a drop-dry method [35, 36]. Schematic diagram of modification of electrode is shown in Scheme 1.

3. Result and Discussion

3.1. Characterization of the Electrodes. The surface morphology of the material was examined with SEM. The SEM images in Figures 2 and 3 show a change in morphology of PANI and Nylon 6,6 nanofibers before and after functionalization with zinc and iron oxide nanoparticles. Figures 2(a) and 2(b) are the SEM images of PANI functionalized with zinc and iron oxides nanoparticles. From the images it is obvious that there is a change in the morphology of PANI after the incorporation of the metal oxides nanoparticles forming clustered and agglomerated films. In Figure 3, the diameter of the functionalized Nylon nanofibers increases and varies from 298 nm to 350 nm compared to the unfunctionalized Nylon 6,6 nanofibers. The FTIR spectra for PANI, Nylon 6,6 nanofibers, and their respective nanocomposites are shown in Figure 4.
Figure 2: The typical SEM images of (a) PANI, (b) PANI/ZnO, and (c) PANI/Fe_3O_4 nanofibers.

Figure 3: The typical SEM images of (a) Nylon 6,6, (b) Nylon 6,6/ZnO, and (c) Nylon 6,6/Fe_3O_4 nanofibers.

Figure 4: The typical FTIR spectra of Nylon 6,6, Nylon 6,6/ZnO, and Nylon 6,6/Fe_3O_4 nanofibers.
Figure 4(a) is the spectra for PANI, PANI/ZnO, and PANI/Fe₃O₄ nanocomposites. The spectra of PANI show the main characteristic peaks at 1580, 1500, 1287, 1147, and 824 cm⁻¹. The bands at 1580 and 1500 cm⁻¹ are attributed to stretching vibrations of N=N ring and N-N ring for benzene rings and quinone rings, respectively, while the bands at 1287 cm⁻¹ correspond to NH bending, and 1147 cm⁻¹ was assigned to C-N stretching of secondary amine amine [37]. The out-of-plane bending vibration of C-H on the 1,4-disubstituted aromatic rings was assigned to the peak at 824 cm⁻¹ [38]. The reduction in the intensity of these PANI characteristic peaks in PANI/ZnO and PANI/Fe₃O₄ nanocomposites and the reduction in PANI broadband intensity at around 2400 cm⁻¹ indicate successful transformation of the PANI molecule to PANI/ZnO and PANI/Fe₃O₄ nanocomposites.

From Figure 4(b) the Nylon 6,6 nanofibers are characterized by O-H stretching vibration around 3950 cm⁻¹, N-H stretching vibration at 3317 cm⁻¹, C-H stretching vibration at 2939 cm⁻¹, and CH₂ bond along with amide peaks at 1652, 1202, 1271, and 1363 cm⁻¹. The peaks at 500 cm⁻¹ reveal the successful incorporation of ZnO and Fe₃O₄ nanoparticles in Nylon 6,6 nanofibers.

Table 1: Values obtained for the reduction potential ($E_{p,a}$), current ($I_{p,a}$), and charge transfer coefficient ($\alpha$) using cyclic voltammetry of the 500 μM lindane in 60:40 methanol/water containing 0.05 M TBAB, with PANI/Zn, Fe(III) and Nylon 6,6/MWCNT/Zn, Fe(III) oxides nanocomposites.

<table>
<thead>
<tr>
<th>Electrodes</th>
<th>$E_{p,a}$ (V)</th>
<th>$I_{p,a}$ (μA)</th>
<th>$\alpha$</th>
</tr>
</thead>
<tbody>
<tr>
<td>GCE/PANI-ZnO</td>
<td>−0.82</td>
<td>98</td>
<td>0.20</td>
</tr>
<tr>
<td>GCE/PANI-Fe₃O₄</td>
<td>−0.73</td>
<td>101</td>
<td>0.23</td>
</tr>
<tr>
<td>GCE/Nylon 6,6/MWCNT/ZnO</td>
<td>−0.58</td>
<td>258</td>
<td>0.25</td>
</tr>
<tr>
<td>GCE/Nylon 6,6/MWCNT/Fe₃O₄</td>
<td>−0.49</td>
<td>23</td>
<td>0.15</td>
</tr>
</tbody>
</table>

was 12 μA on the bare GCE, while on the modified electrodes, as shown in Table 1, there was an increase in reduction current for lindane. The comparative response at the modified electrodes studied reveals that the reduction peak current is higher at Nylon 6,6/MWCNT/Fe₃O₄, Nylon 6,6/MWCNT/ZnO modified electrode and more positive potential than the bare electrode which may be due to the enhanced diffusion of lindane molecules through the micropores of Nylon 6,6/MWCNT/Fe(III) oxides nanofibers [39, 40]. For the cyclic voltammetric response of 500 mM lindane at the modified electrodes, after 10 cycles of multiscans, only a small decrease in peak current is observed. This also further confirms that the reduction process of lindane at the modified electrodes is diffusion controlled. If the adsorbed species on the electrode surface completely block the mass transport, it is then expected that there may be disappearance of the peak after multiscans. Therefore the absence of adsorption processes during the electroreduction of lindane at these modified electrodes is expected to improve the sensitivity of the sensor compared to the bare GCE sensor.

The cyclic voltamogram scan rate study of the modified GCE electrodes of PANI/Zn, Fe(III) and Nylon 6,6/MWCNT/Zn, Fe(III) oxides nanofibers was studied. Figures 6 and 7 show the scan rate study for Nylon 6,6 and PANI based nanocomposites, respectively. The linear relationship between the peak current and square root of the scan rate as shown in Figures 6 and 7 obtained for modified GCE electrode of PANI/Zn, Fe(III) and Nylon 6,6/MWCNT/Zn, Fe(III) oxides nanocomposites shows that lindane undergoes a diffusion controlled reduction process. A high slope value is obtained which also indicates that the reduction process is electrotransfer (ET) [41]. The number of electrons transferred during the reduction of lindane is calculated from the Randles-Sevcik equation for irreversible reduction processes by substituting the diffusion coefficient value of 0.89 × 10⁻⁵ cm² s⁻¹ for lindane reported in the literature [42]:

$$I_p = \left(2.99 \times 10^5\right)a^{0.5}n^15n^{15}CD^{0.5}v^{0.5},$$

where $n$ is the number of electrons transferred, $a$ is the transfer coefficient, $D$ is the diffusion coefficient in cm²/s⁻¹, $C$ is the concentration in mol cm⁻³, $v$ is the scan rate in V/s⁻¹, and $A$ is the area of the electrode in cm². In order to get information on the rate determining step, the peak potential, $E_p$, is proportional to log $v$ (not shown). The slopes of $E_p$ versus log $v$ whose relationship is shown by (2) for the
different electrodes were 0.148, 0.131, 0.120, and 0.202 V for electrodes GCE/PANI-ZnO, GCE/PANI-Fe$_3$O$_4$, GCE/Nylon 6,6/MWCNT/ZnO, and GCE/Nylon 6,6/MWCNT/Fe$_3$O$_4$, respectively. The value of $\alpha$ is calculated from the Tafel equation (3). From the slope of the Tafel plot the experimental electron transfer coefficient $\alpha$ is determined. The high value of $\alpha$ is a clear evidence that a large double layer effect is present for the charge reactants [43]:

\[ E_p = \left( \frac{b}{2} \right) \log \left( \frac{v}{\text{mVs}^{-1}} \right) + \text{constant} \]  

(2)

slope = \frac{1.15RT}{F\alpha}, \quad (3)

where $T$ is the absolute temperature, $R$ is the gas constant, and $\alpha$ is the so-called charge transfer coefficient, the value of which must be between 0 and 1 as shown in Table 1. Also from the table the smaller values of $\alpha$ further support the fact that electrotransfer (ET) process and bond breaking are concerted [44]. Therefore the number of electrons ($n$) involved in the reduction process is determined to be 6 which also supports the earlier reports for benzene formation [41].

### 3.3. Electroanalysis of Lindane

To improve the sensitivity of the proposed method in detection of lindane, square wave voltammetry (SWV) has been used. From Figure 8, it is found that the electrochemical reduction peak current is
Figure 7: Effect of scan rate (25–300 mV/s) on the cyclic voltammograms of (a) GCE/PANI/ZnO and (b) GCE/PANI/Fe₃O₄ in 500 µM lindane in 60:40 methanol/water containing 0.05 M TBAB. (c) and (d) are the plots of current (I) against square root of scan rate $\sqrt{\nu}$ (mV/s) for GCE/PANI/ZnO and GCE/PANI/Fe₃O₄, respectively.

TABLE 2: Values obtained for the concentration study of 500 µM lindane in 60:40 methanol/water containing 0.05 M TBAB, at modified PANI/Zn, Fe(III) and Nylon 6,6/MWCNT/Zn, Fe(III) oxides nanocomposites electrodes using square wave voltammetry.

<table>
<thead>
<tr>
<th>Electrodes</th>
<th>Concentration range (mol/L)</th>
<th>LoD (nM)</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>GCE/PANI-ZnO</td>
<td>$9.9 \times 10^{-12}$ - $5 \times 10^{-6}$</td>
<td>239.0</td>
<td>0.9133</td>
</tr>
<tr>
<td>GCE/PANI-Fe₃O₄</td>
<td>$9.9 \times 10^{-12}$ - $5 \times 10^{-6}$</td>
<td>44.7</td>
<td>0.8277</td>
</tr>
<tr>
<td>GCE/Nylon 6,6/MWCNT/ZnO</td>
<td>$9.9 \times 10^{-12}$ - $5 \times 10^{-6}$</td>
<td>51.0</td>
<td>0.8349</td>
</tr>
<tr>
<td>GCE/Nylon 6,6/MWCNT/Fe₃O₄</td>
<td>$9.9 \times 10^{-12}$ - $5 \times 10^{-6}$</td>
<td>32.0</td>
<td>0.9733</td>
</tr>
</tbody>
</table>

proportional to lindane concentrations in the range of $9.9 \times 10^{-12}$ mol/L to $5 \times 10^{-6}$ mol/L for modified PANI/Zn, Fe(III) and Nylon 6,6/MWCNT/Zn, Fe(III) oxides nanocomposites electrodes, respectively. The detection limit was calculated based on the relationship $\text{LoD} = 3.3\delta/m$ [45], where $\delta$ is the relative standard deviation of the intercept of $y$-coordinates from the line of best fit and $m$ is the slope of the same line. The respective detection limits for the electrodes are shown in Table 2. These values are found to be lower than LoD values reported at NiCo₂O₄ and cellulose acetate modified glassy carbon electrode [46, 47] but eight times higher than the LoD report on CuO/MnO₂ hierarchical nanomicrostructures by Kumaravel et al. [39].

3.4. Interference Studies. The interference study was carried out using cyclic voltammetry. The interfering substances considered in this work may be present in water or soil from industrial areas and agricultural land [46]. The possibility of the interference of several inorganic ions in the reduction of the signal of lindane was studied by analyzing...
I(\mu A) versus (Ag/AgCl, sat’d KCl)

-1.4 -1.2 -1.0 -0.8 -0.6 -0.4 -0.2 0.0 0.2 0.4 0.6 0.8 E(V)

9.9 \times 10^{-12} \text{ mol/L}
9.9 \times 10^{-11} \text{ mol/L}
3.3 \times 10^{-10} \text{ mol/L}
1.0 \times 10^{-8} \text{ mol/L}
5.0 \times 10^{-6} \text{ mol/L}

(a)

(b)

3.5. Real Sample Analysis. Real sample analysis was carried out by using Nylon 6,6/MWCNT/Zn, Fe(III) oxides nanocomposites sensors to determine the concentration of lindane in tap water samples. Samples containing different amounts of lindane were prepared in 60:40 (v/v) methanol-tap water (20 mL). The values of recovery were in the range from 99% to 102%, suggesting the accuracy of Nylon 6,6/MWCNT/Fe$_3$O$_4$ and Nylon 6,6/MWCNT/ZnO nanofibers-based sensors.

4. Conclusions

This work describes the electron transport and electrocatalytic properties of chemically synthesized metal oxide nanoparticles (Fe$_3$O$_4$ and ZnO) supported on polyaniline and multiwalled carbon nanotubes-Nylon 6,6 nanofibers platforms. It is shown that PANI/Zn, Fe(III) and Nylon 6,6/MWCNT/Zn, Fe(III) oxides nanocomposites modified electrode offers a high sensing current and lesser reduction potential for lindane than the bare GCE electrode. The Nylon 6,6/MWCNT/Zn, Fe(III) oxides nanocomposites modified electrode gave a lower limit of detection for lindane than the PANI/MO nanocomposites modified electrodes. It was also highly stable and reproducible with respect to time, so that the electrode can be used for multiple detection of lindane. Cyclic voltammetry and square wave voltammetry were used as sensing techniques.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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