Synthesis of graphene-Mn, O nanocomposite as electrode sensors of profenofos

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Abstract. Synthesis and characterization of of Graphene-Mn nanocomposites as Pesticide sensor electrodes have been carried out. This study aims to determine the method for synthesizing and characterizing the products of Graphene-Mn nanocomposite synthesis. Modification of Graphene using Mn nanoparticles for profenofos analysis with cyclic voltammetry. This research using with the Modified Hummers method and the liquid phase adsorption method followed by hydrothermal. Characterization of Graphene-Mn oxide nanocomposites are displayed using XRD, SEM-EDX and FTIR. The XRD results showed that there are peaks of MnO (30.57, 44.39), MnO2 (34.14), and MnO (39.85, 57.85, 68.94). SEM-EDX analysis reveals the surface of graphene sheets containing aggregates in shape small particles attached to graphene sheets. The results of the EDX analysis consisted of Mn 6.26%, C 53.60% and O. 15.45%. The FTIR test indicates the presence of Mn-O bonds in wave number 615.29 cm⁻¹ and other functional groups, such as O-H, C-O, C = C, and C-H.

Electrodes with the best composition at pH 6 will be used to detect pesticides profenofos with the Cyclic Voltammetry (CV) method at a scate rate of 0.05 V / s. The detection limit obtained from graphane-Mn measurements on profenofos is 0.000667 M. and the repeatability of graphane-Mn performance is 0.0131. The age of Graphene-Mn electrodes in analyzing profenofos pesticides is 17 days. The selectivity test using Pb (NO3)2 0.1M and Phenol 0.1M disrupting compounds occurred in a negative direction.

1. Introduction

Detection of levels of pesticides in the environment is needed to minimize the negative effects of the dangers of over-use of pesticides. An alternative method that the electroanalysis method, in this case voltammetry is one of the most sensitive analytical methods that has high selectivity low detection limits, and easy sample preparation and measurement [1]. The performance of the voltammetry method is strongly influenced by the working electrode material [2]. The working electrode is composed of a mixture of carbon materials such as graphite.

Graphene is a monolayer (single layer) with two dimensional shapes of carbon which has a hexagonal structure resembling a honeycomb in sheet form [3]. The sheets were bound by Van der Waals bonds with a distance between 0.335 nm. Graphene has an atomic distance of 0.142 nm and is bound by covalent bonds [4].

Graphene is highly conductive with charge carrier mobility of up to 200,000 cm²V⁻¹S⁻¹, electrical conductivity of up to 5,300 Wm⁻¹K⁻¹ [5]. With the advantages of its properties, graphene has the potential to be developed as an electronic device component [6] and it is interesting to form composite materials used as electrode material, however, pure graphene has poor pore...
affinity, low efficiency[7]. To overcome the poor pore affinity of graphene, a doping agent is needed to fix it.

Currently, the support of carbon and metal materials has evolved with the Liquid Phase adsorption (LPA) method combined with the hydrothermal method. The LPA method is a method that can be used for nanoparticle production through liquid phase adsorption. The development in the LPA method aims to store the active ingredients in nanocarbons[8]. In addition to the advantages that have been described, there are also disadvantages to the liquid phase adsorbs method, which is that it cannot form oxides on the metal ions used so it needs to be continued with the hydrothermal method.

Hydrothermal synthesis is one of the techniques of crystallizing from high temperatures in aqueous solutions at high pressure[9]. Some of the advantages of the hydrothermal method are that it produces particles with high crystallinity, high purity, and homogeneous particle size distribution [10] base on the description in the research, the pesticide Profenofos testing will be carried out using graphene-Mn electrodes. The presence of Mn as a doping metal is expected to improve graphene performance on the profenofos analysis.

2. Methodology

2.1. Tools
The tools used in this study, namely DY2100B potentiostat, platinum electrodes, Ag/AgCl comparison brushless Centrifuge MPW-350) X-Ray Diffraction (Shimadzu 6000), Fourier Transform Infra Red (FTIR), (Shimadzu 8400) and Scanning Electron Microscopy (HITACHI SU 3500).

2.2. Materials
The materials used in this study are graphite, Manganese Stone (Origin from Buton Island), potassium permanganate (KMnO₄), sulfuric acid (H₂SO₄) 98%, hydrogen peroxide (H₂O₂), Sodium Nitrate (NaNO₃) hydrochloric acid (HCl) 37%, Pb(NO₃)₂, Phenol, Citric Acid, Sodium Citrate, liquid paraffin Copper wire, 90% profenofos, ethanol. All compounds were obtained from Sigma Aldrich.

2.3. Graphite Oxidation
Graphite oxide was synthesized with the modified Hummer method. The synthesis process uses graphite powder, KMnO₄, NaNO₃, and H₂SO₄ as the material base. The synthesis process begins with stirring 2 g graphite powder and 4 g NaNO₃ with 98 mL 98% H₂SO₄ for 4 hours at high speed in an ice bath. After the stirring process runs for 1 hour, as much as 8 g KMnO₄ and 4 g NaNO₃ begin to be added gradually. Furthermore, the stirring process continued at 25 °C for 24 hours. Then 200 mL of distilled water is added gradually to the solution and stirred for about one hour or until the solution is homogeneous. After the solution is homogeneous, 15 mL of hydrogen peroxide (H₂O₂) is added gradually while stirring. After that, the solution is separated between the solid and liquid phases by using a centrifuge with a speed of 3000 rpm for 1 hour. The solid phase that has been separated from the liquid phase is washed using 10 mL 37% HCl and distilled water several times until the pH of the solution is neutral.

2.4. Leaching Natural Mangane
Mangane leaching was conducting by weighing 20 grams of mangane rocks that have been mashed and dissolved into 50 mL HCl (37%). Then stirrer for 3 hours which is kept constant 70 °C, the stirring solution is filtered using filter paper to extract the final volume, then the residue from the solution is weighed.

2.5. Synthesis of Graphene Oxide (GO)
0.4 grams of graphite oxide are dissolved in 100 mL distilled water until homogeneous. After being homogeneous, the solution is sonicated for 3 hours. Graphite Oxide will undergo a process of exfoliation into graphene oxide.
2.6. Synthesis of Graphene doped (Mn)
Synthesis of GO doped Mn with a variation of 8: 5 was carried out by reacting GO 320 mL (0.8 gram) solution dried to a volume of 100 mL and added 1.75 mL of manganese filtrate to form a precipitate. After that, dissolved graphene oxide was cultivated with a frequency of 53 kHz for 30 minutes. Followed by a hydrothermal process at a temperature of 180 °C for 24 hours. Graphene/Mn formed was dried at 110 °C for 24 hours, dried and weighed Graphene-Mn formed.

2.7. Manufacturing of Work Electrodes
Manufacturing of Work Electrode is carried out by adding Graphene-Mn powder and paraffin little by little until the texture formed the paste. The mixture is inserted into the electrode body and allowed to stand for 2 days until it solidifies. After that, the electrode surface is rubbed until smooth, flat, and glossy using sandpaper.

2.8. Electrode Test in Analyzing Profenofos pesticides

![Electrode Test](image)

Figure 1. Design of electrode measurements by voltammetry

3. Results and Discussion
3.1. Synthesis Of Graphene-MnO Nanocomposite
Analysis of graphite and graphene crystal structure conducting with X-Ray Diffraction at an angle of 2θ = 20-80° and λ Cu-Kα = 1.54060 Å. Based on Match software analysis that has been adapted to JCPDS (Joint Commite Powder of Diffraction Standard) No. 03-065-6212. From the result, it observed the comparison of XRD patterns of graphite oxide and graphene, the peak of graphite that appears in 26.5149° with dspacing 3.359 Å. In graphene oxide there is a new peak which is 23.40° with dspacing 3.857 Å. The dspacing value which is getting bigger shows that graphite has been reduced to graphene. In addition, dspacing graphene values greater than graphite indicate that there is peeling of the graphite layer into a single layer graphene, as showed in the following figure.

![XRD Patterns](image)

Figure 2. XRD patterns of graphite oxide and graphene synthesized

Glosarium:
1. Ag / AgCl comparison electrodes
2. Auxiliary Electrodes Pt
3. Working Electrodes
4. Test Solution
In Figure 2 showed the peak intensity of graphite oxide with an angle of 26.5149° with d-spacing 3.359 Å. In graphene there is a new peak which is 23.04° with 3.857 Å d-spacing. Distance and left angle shifting occur when graphite oxide turns into graphene caused by the loss of phenol, ketone, epoxy carboxyl and carbonyl groups [11]. From previous studies, the value of the peak of graphite in angle 12.43° with d-spacing 7.110 Å and the peak of graphite oxide is 26.26° and d-spacing 3.390 Å [12]. The d-spacing value which is getting bigger showed that graphite has been reduced to Graphene.

Figure 3. XRD pattern of graphene doped by manganese

Figure 3 showed the results of the graphene-Mn$_x$O$_y$ XRD pattern. Based on the results, it can be seen that there is a diffractogram for peaks of $2\theta = 23.02°$ and $44.48°$ which is a typical peak of graphene oxide which is marked by the presence of C atom. However, the peak is not so visible, this is due to carbon reacted with metal oxides will coincide and tend to be covered by metal oxides. This is consistent with research conducted by [13, 14] which states that the highest intensity in graphene oxide is at the miller index (001), where these results are in accordance with the standard joint commite powder of diffraction (JCPDS-41-1487). The results of XRD characterization of graphene doped manganese can be seen at angles $2\theta = 30.57$ and $44.39°$ with d-spacing 2.9216 Å and 2.0390 Å are Mn$_3$O$_4$ crystal patterns, angles $2\theta = 34.14°$ with d spacing 2.6357Å are MnO$_2$ crystal patterns, where as at angles $2\theta = 34.14, 39.85, 57.85, 68.94°$ with successive d spacing values 2.260 Å, 1.5917 Å, 1.3610 Å, respectively are MnO crystal patterns. From the results of FTIR analysis, graphene oxide has functional groups O-H, C-H, C = O, C = C, and C-O, where as in graphene Mn$_x$O$_y$ oxide has functional groups O-H, C = C, C-H, C=O and Mn-O.

Table 1. Graphene-MnxOy FTIR spectrum data.

| Functional groups | Graphene Oxide | Graphene/Mn$_x$O$_y$ |
|-------------------|---------------|---------------------|
| O-H               | 3417.86       | 3414                |
| C-H Aliphatic     | 2850, 792918,30 | -                   |
| C-H               | -             | 1402.25             |
| C=C Aromatic      | 1571.99       | -                   |
| C=O               | 1124.50       | 1116.78             |
| C=O               | 1720.50       | -                   |
| Mn-O (400-700 cm$^{-1}$) | -     | 615.29              |
Based on the functional groups that are identified there are differences in the peak absorption area of graphene Oxide and graphene-Mn, the peak of graphene Oxide that has been lost after being synthesized include C=C aromatic stretching 1571.99 cm$^{-1}$, CH Aliphatic stretching 2850.79-2918.30 cm$^{-1}$, C=O stretching 1720.50 cm$^{-1}$. With the loss of the functional group it is suspected that graphene Oxide has been successfully reduced to graphene, and the presence of Mn-O stretching absorption area indicates that Mn$_x$O$_y$ was doped successfully. The results of characterization using SEM provide information about the shape of the surface morphology and determine the elements contained in nanocomposites. Characterization using SEM was carried out to determine the shape of the surface morphology of graphene oxide and graphen-Mn$_x$O$_y$ that has been synthesized.

![Figure 4](image)

Figure 4. (a) graphene oxide at 10000x magnification (b) graphene-MnxOy magnification at 10000x

From the results of SEM photographs in figures (a) and (b) there are differences in the surface of graphene. It can be seen in figure (a) that the surface of graphene oxide has been exfoliated and graphite structures are destroyed due to sonication. Previous research conducted by Azizah and Susanti[15] suggested that the morphology of graphene oxide had turned into sheets consisting of several layers, this means that the sample results of SEM characterization in Figure (a) were GO sheets. Figure (b) shows the visible interaction of foreign particles that have different morphology from the sheet, it can be assumed as Mn$_x$O$_y$ particles deposited on graphene sheets in the form of aggregates that are widely spread on the surface. This shows that there are interactions between the two particles. Formation and transformation of the crystalline phase of manganese oxide nanoparticles consisting of agglomeration of much smaller manganese granules seen. Interactions that occur in Mn ions are thought to be chemical interactions formed by phi bonds ($\pi$) where these bonds are the cause of electron exchange. Here is a description of the interactions in graphenenanocomposites and Mn ions.
These results were also confirmed by the results of the EDX analysis. Analysis with EDX can provide information on components of Mn doped graphene both qualitatively and quantitatively. The qualitative analysis aims to identify the constituent elements of Mn doped graphene, while the quantitative analysis aims to determine the percentage and amount of element content contained in the Mn doped graphene nanocomposite.

From the figure 6 showed the presence of C, O, and Mn atoms contained in nanocomposites. The existence of these atoms indicates that the Graphene-MnxOy composite has been successfully synthesized. The detected impurities in the form of Cl as much as 6.17% are thought to originate from the solvent used, and the elements F, S, Ba are thought to originate from manganese which is dissolved in the leaching process.
3.2. Comparative Test of NaNO\textsubscript{3} Electrolyte Solution and Profenofos with Graphene-Mn Electrode

The determination of Profenofos has been carried out using Graphene-Mn as work electrode with the best composition variation of 8:5, the measurement parameters used are: init E(V) = -0.8; high E(V) = 0.8; low E(V) = -0.8; scan rate (V/s) = 0.05; and sens (A/V): 10\textsuperscript{-3}. These parameters will be used until the final test of sample testing using the cyclic voltammetry method. The use of NaNO\textsubscript{3} as supporting electrolyte based on research conducted by Salim\textsuperscript{[16]} NaNO\textsubscript{3} is a supporting electrolyte that serves to maintain the strength of ions in solution and reduce the flow of migration. A comparison of the peak current between the profenofos test solution and the NaNO\textsubscript{3} electrolyte needs to be done to determine whether the peak current generated is from the analyte or electrolyte. The measurement results was showed in Figure 7.

![Figure 7. Voltammogram comparison of NaNO\textsubscript{3} with profenofos](image)

Based on the measurement, it can be seen that there is a fairly large current difference between NaNO\textsubscript{3} and profenofossolutions. NaNO\textsubscript{3} electrolyte solution produces a peak current (Ipa) of 11.9 µA and a peak (Ipc) of -22.6 µA. Good supporting electrolyte solution that is not reacting with analyte and stable\textsuperscript{[17]}. While the Profenofos solution produces an anodic peak current (Ipa) of 47.1 µA and a Cathodic potential (Ipc) of -47.2 µA at a potential of 0.2 V. Determination of the optimum pH was conducted. The best composition is the ratio of 8: 5 to the standard solution of Profenofos 0.1M. The citrate buffer used to determine the optimum pH is pH 3, 4, 5 and 6, the reason for using the buffer at that pH with acidic conditions is because according to Nurpadillah\textsuperscript{[18]} the nature of profenophos is less stable under alkaline conditions. The effect of pH was studied by comparing the voltammogram measured by analyte solution with varied pH conditions. Determination of the optimum pH is indicated by the highest peak current on the voltamogram.
Based on the measurement results, the optimum peak current showed in the addition of a buffer with pH 6, the optimum pH condition is suspected to be profenofos in the form of free ions in large amounts, so that there will be a pH 6 equilibrium condition in the analyte solution. Furthermore measurements at pH 3 and 4 decreased current, because at that pH the condition of the analyte solution is too acidic, with the number of protons (H⁺) in the solution being more so that the resulting potential will be disturbed, by the presence of more H⁺ ion activity and making profenophos cannot contact properly on the electrode surface.

### 3.3. Determination of Linearity and Detection Limit on Graphene-Mn Electrode

The linear concentration range of the profenofos measurements is determined by making a series of standard solutions profenofos with a concentration of $10^{-6}$, $10^{-5}, 10^{-4}, 10^{-3}, 10^{-2}$ M in 0.1 M NaNO₃. The linear equation was determined by plotting between $I_{pa}$ and the concentration of standard solution. Based on the measurement results it can be seen that the higher the concentration of the test solution the greater the value $I_{pa}$ obtained. This is caused by the higher concentration, the more ions that accumulate on the surface of the electrode so that current the resulting yield is also higher measurement results are shown in Figur
The purpose of making this linear curve is to find out a good working range of standard linearity in the measurement of profenophos. This concentration range has a linear correlation between the concentration of profenophos and the current produced, with an $R^2$ of 0.96. Based on the opinion of Fisher, in Azizah [19] which states that the level of relationship correlation value $R^2$ with a range of 0.80 to 1.0 has a very strong linear relationship.
3.4. Repeatability test

The repeated measurements were carried out 11 times measurement of profenofos solution. Then, a histogram are made between the repetition and the peak current generated for each measurement. Based on the data obtained in figure 10, Graphene-Mn can be said to be stable in analyzing profenophos.

![Figure 10. The histogram of repeated measurement](image)

3.5. Electrode Age Test

Electrode age test conducted to determine the stability of electrodes in a long time that is still feasible to use to analyze an analyte[20]. The age test of the electrodes was measured by Graphene-Mn on profenofos for 25 days with a time span of measurements once a day. The measurement results showed in Figure 11.

![Figure 11. Histogram of electrode age](image)

Based on figure 11, on the first day to the 17th day, electrodes are still in a stable condition which is marked by the peak current produced high and relatively the same. On the 18th day to the 21st day peak current decreased, measurements on the 22nd and 25th electrodes produced a very low peak current. The decrease in peak current caused by the use of electrodes carried out continuously can cause the diffusion layer to thicken the surface of the electrode due to contact with the test solution.
continuously, according to [21] the decrease in peak current results in electron transfer taking place slowly and the peak current produced decreases. Decreased electrode response was also caused because electrodes can decay and are unstable at room temperature after being stored for a long time [22].

3.6. Addition of Disruptors
The determination of Graphene-Mn selectivity was carried out on voltammetry cells which contained Profenofos's solution with citrate buffer pH 6 with a concentration of 0.1 M then the addition of Phenol with a concentration of 0.1 M as a confounding compound. The measurement results are shown in Figure 12.

![Figure 12. The profenofos voltammogram with phenol compounds as addition](image)

From Figure 12 showed that the measurement of the current that has been added to the Phenol Disturbance Compound produces a greater negative potential. This change is caused by a high concentration of phenol and the closure of the electrode surface by phenol compounds in addition presumably due to the contact reaction between the profenofos test compound and the intruding compound so that the measurement results shift the profenofos potenti.

4. Conclusion
Based on the results and discussion described, it can be concluded as follows
1. Graphene-Mn₃O₄ synthesis has been carried out with Hummer's modified method with liquid phase adsorption and continued with hydrothermal, SEM characterization results at 1000x magnification showed manganese oxide particles on graphene sheets. EDX results contain 53.60 %. C, O as much as 15.45% and Mn 6.26%. XRD doped graphene results showed the peaks of Mn₃O₄, MnO₂ and MnO, the FTIR spectrum was also confirmed Mn-O interaction at the wave number 615.29 cm⁻¹ and loss of some functional groups after doping indicates that graphene has been successfully reduced by mangane ions.
2. Cyclic Voltammetry Results with Innit Parameters of -0.8, high 0.8, low -0.8 and sens 10⁻³ showed peaks of Ipa and Ipc with NaNO₃ supporting electrolytes 0.1M, addition of pH 6 buffer indicates optimum conditions. The detection limit obtained was 6.67x10⁻⁴M and the repeatability was indicated by a Horwitz Ratio (HorRat) value of 0.11. The optimum measurement stability in analyzing profenofos is 17 days and there is a significant influence on profenofos analysis when added to a confounding compound as evidenced by the emergence of a typical cathodic peak current and a potential negative shift in the addition of phenols.
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