DEVELOPMENT OF NANO TITANIUM DIOXIDE MODIFIED CARBON PASTE ELECTRODE FOR DETERMINATION OF CHLORAMPHENICOL IN VANAME SHRIMP (*Litopenaeus vannamei*) POND WATER BY CYCLIC VOLTAMMETRY

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Received: October 13, 2023. Accepted: : November 27, 2023. Published: November 30, 2023

Abstrak: Chloramphenicol is an antibiotic that has activity against aerobic, anaerobic, and fungal bacteria and is often used in vaname shrimp farming pond water. Behind its usefulness, chloramphenicol has side effects for health, such as aplastic anemia, bone marrow suppression, stomach intestinal disorders, and optical and peripheral neuropathy. This study aims to determine the ability of carbon paste electrodes modified with nano titanium dioxide to detect chloramphenicol levels in vaname shrimp pond water by cyclic voltammetry. This research has successfully made an electrochemical sensor as a nano titanium dioxide-modified carbon paste electrode to detect chloramphenicol in vaname shrimp pond water samples. Nano titanium dioxide used as a modifier in carbon paste electrodes was characterized by XRD (X-Ray Diffraction) and FTIR (Fourier Transform Infrared), while the study of electrochemical characteristics using cyclic voltammetry method. The electrochemical sensor that has been made is a nano titanium dioxide-modified carbon paste electrode in vaname shrimp pond water samples. The electrochemical sensor that has been made is a nano titanium dioxide-modified carbon paste electrode that can detect chloramphenicol in vaname shrimp pond water wethod.

Keywords: Chloramphenicol, Carbon Paste Electrode, Nano Titanium Dioxide, Cyclic Voltammetry

INTRODUCTION

Indonesia is a maritime country dominated by its water area, which accounts for 65% of the total area of Indonesia. It has a coastline of 81,000 km [1]. The area of Indonesia's inland waters and archipelagic waters is 3,110,000 km² with a coastline length of 108,000 km [2]. Therefore, it can be said that Indonesia has great potential in the fisheries sector. One of them is the aquaculture business in the form of vaname shrimp (*Litopenaeus vannamei*). Vaname shrimp has much demand domestically and abroad [3].

Lamongan Regency is the largest producer of vaname shrimp in East Java, and vaname shrimp is the primary commodity in this region. The vaname shrimp commodity has several advantages: high productivity, easier cultivation, resistance to disease, shorter maintenance time because the growth is relatively fast, and stronger resistance thaniger shrimp [4]. According to [5], the Lamongan Regency is one of the regencies with a coastline length of 47 km and a pond area of 932.29 ha. Based on data from the Lamongan Regency Fisheries Service in 2020, the production of *Litopenaeus vannamei* called white shrimp or vaname cultivation, reached up to 16,194.03 tons [5].

Vaname shrimp farming requires antibiotics to prevent diseases in vaname shrimp, such as diseases caused by *Vibro Harveyi* bacteria and white spot disease caused by WSSV (*White Spote Syndrome Virus*). This virus can cause shrimp death by up to 100% [6, 7]. One type of antibiotic used in vaname shrimp farming is chloramphenicol.

Chloramphenicol is an antibiotic for aquaculture animals such as vaname shrimp that has

activity against aerobic anaerobic bacteria and fungi [8]. According to Badan POM in 2023, based on the Regulation of the Indonesian Minister of Health Number 33 of 2012 concerning Food Additives (in Indonesian abbreviated as BTP), chloramphenicol is a type of food additive prohibited in Indonesia. Exposure to humans can cause aplastic anemia, bone marrow suppression, stomach and intestinal disorders, and optical and peripheral neuropathy [8]. This is why chloramphenicol residues are not allowed in food animal products. Chloramphenicol analysis can use the cyclic voltammetry method.

Cyclic voltammetry is an electroanalytical method widely used to obtain information about electrochemical reactions (analyzing currentpotential curves) in solution. [9, 10]. The main advantages of electrochemical reactions are simple research procedures, fewer reagents, and an analyte detection limit in concentrations of mg/L to ppb [11].

The voltammetry cell consists of three electrodes: the auxiliary electrode, the comparison electrode, and the working electrode. The working electrode has a vital role in the voltammetry method because the working electrode is where the oxidation-reduction reaction occurs [12]. Three constituent electrodes in a voltammetric cell is immersed in a solution. The first electrode is the working electrode, reference or comparison electrode, and auxiliary electrode [13, 14, 15, 16]. The working electrode used in this study is a nano titanium dioxide-modified carbon paste electrode.

Carbon paste electrode (EPK) is a material used as a current conductor in electrolysis cells

[10]. Carbon paste electrodes (EPK) aim to accumulate several lyophilic ions by discharge analysis in voltammetry and potentiometry. Under acidic conditions, this paste liquid functions as an anion exchanger due to several functional groups on the electrode reactivity, followed by increasing the composition of the paste liquid and reducing the electron transfer rate [17]. The main advantages of carbon paste electrodes in the analysis process are that there is no risk of mechanical damage to the electrode material, which is very beneficial for use during the research process, it is easy to renew, the electrode surface is easy to modify, and reproducible [14]. This study used nano titanium dioxide (EPKN) modified carbon paste electrodes to analyze chloramphenicol levels in vaname shrimp pond water (Litopenaeus vannamei).

Research on chloramphenicol levels in vaname shrimp pond water has been conducted with several methods, such as HPLC (High-Performance Liquid Chromatography) [18]. The disadvantages of the HPLC method are the short working life (related to column life) and the high flow rates. It requires high costs for purchase. In addition, the disposal of solvents with high purity [19].

The development of nano titanium dioxide modified carbon paste electrode was carried out because nano titanium dioxide has good dispersing properties and fast electron transfer ability. The development and application of chemically modified carbon paste electrodes have received considerable attention due to their relatively wide anodic potential range (in this study, a -1.5 to +1.5 V anodic potential range was used), low residual current, ease of manufacture, large surface area, and good dispersion properties [20].

RESEARCH METHODS

This type of research is experimental research in manufacturing carbon paste electrodes (in Indonesian, abbreviated as EPK). The carbon paste electrode is a nano titanium dioxide-modified carbon paste electrode or can be called EPKN. The nano titanium dioxide-modified carbon paste electrode is used to analyze chloramphenicol levels in vaname shrimp pond water (*Litopenaeus vannamei*).

The tools used in this study consist of Voltammetry Instrumentation (797 VA Computrace), UV-Vis Spectrophotometry (Shimadzu UV-1800), XRD (X'Pert PRO PANalytical), FTIR (Thermo scientific nicolet iS10), pH meter (Eutech Instruments), analytical balance (Ohaus), 50 mL beaker (Iwaki), 100 mL beaker (Pyrex), 250 mL beaker (Iwaki), 100 mL volumetric flask (Iwaki), 250 mL volumetric flask (Iwaki), 10 mL measuring cup (Iwaki), 100 mL measuring cup (Duran), ball pipette, 5 mL measuring pipette, 10 mL measuring pipette, spray bottle, 200 μ L micropipette (Dragon Lab), blue tip, 100 mL glass vial, 500 mL glass vial, stirrer, magnetic stirrer, centrifuge (Eppendorf 5810),

spatula, glass stirrer, alligator and cable tongs, sandpaper, scissors, copper wires, watch glass, Petri dish, and oven (Corsair Heating and Catering Ltd.).

The materials used in the study were vaname shrimp pond water, chloramphenicol (Himedia CMS218-5G), carbon powder, paraffin oil (Brataco), sodium dihydrogen phosphate (NaH₂PO₄.2H₂O) (Sap Chemicals) 0.1 M, sodium hydrogen phosphate (NaH₂PO₄. 2H₂O) 0.1 M (Supelco), KCl p.a, distilled water (Cims), Ag/AgCl electrode as comparison electrode, platinum electrode as auxiliary electrode, and 99% anatase nano titanium dioxide (Hongwu Nanomaterial) as working electrode.

Sample preparation

Samples were taken from vaname shrimp pond water with potential chloramphenicol presence in Wangen Village, Glagah District, Lamongan Regency.

Preparation of phosphate buffer solution

A total of 3.44975 grams of stock solution A (NaH₂PO₄.2H₂O) 0.1 M was dissolved in 250 mL of distilled water, and 3.549 grams of stock solution B (Na₂HPO₄) 0.1 M in 250 mL of distilled water. Making phosphate buffer solutions of various pH is done by mixing stock solution A with stock solution B [13, 10]

Preparation of unmodified carbon paste electrode

Carbon powder of 0.5 grams and paraffin oil of 0.5 grams were mixed in a cup. The carbon paste mixture was stirred until homogeneous. In making the electrodes, 10 cm long copper wires were given an upper limit of 2 cm and a lower limit of 0.5 cm. The insulator was peeled off, and the copper wire was sanded until smooth and shiny. The bottom of the electrode body is given a minor plastic pipe cut to protect the carbon paste so that there is no release. Then, the homogeneous carbon paste mixture is manually inserted using a spatula [13, 10].

Preparation of nano titanium dioxide modified carbon paste electrode

Carbon powder of 0.5 grams and paraffin oil of 0.5 grams were mixed in a cup. The carbon paste mixture was stirred until homogeneous. In making the electrodes, 10 cm long copper wires were given an upper limit of 2 cm and a lower limit of 0.5 cm. The insulator was peeled off, and the copper wire was sanded until smooth and shiny. The bottom of the electrode body is given a minor plastic pipe cut to protect the carbon paste so that there is no release and then the homogeneous carbon paste mixture is inserted using a spatula manually [13, 10]. J. Pijar MIPA, Vol. 18 No. 6, November 2023: 986-993 DOI: 10.29303/jpm.v18i6.5844

Preparation of optimum pH

The working electrode will be the carbon paste electrode modified with nano titanium dioxide with the best composition. The voltammetric cell is filled with 10 mL of analyte solution added with 10 mL of KCl solution with a concentration 50-100 times greater than the analyte and 5 mL of phosphate buffer solution pH 5, 6, 7, 8. After that, current measurements are made at a potential of -1.5 V to +1.5 V with a deposition time of 10 seconds and a scan rate of 0.05 V/s [13, 10].

Determination of the best deposition time

The working electrode will be the carbon paste electrode modified with nano titanium dioxide with the best composition. The voltammetric cell is filled with 10 mL of analyte solution added with 10 mL of KCl solution with a concentration 50-100 times greater than the analyte and 5 mL of phosphate buffer solution of optimum pH. Then, current measurements were taken at potentials of -1.5 V to +1.5 V with deposition times of 10, 20, 30, 40, and 50 seconds and a scan rate of 0.05 V/s [13, 10].

Determination of the best scan rate

The working electrode will be the carbon paste electrode modified with nano titanium dioxide with the best composition. The voltammetric cell is filled with 10 mL of analyte solution added with 10 mL of KCl solution with a concentration 50-100 times greater than the analyte and 5 mL of phosphate buffer solution of optimum pH. Then, current measurements were carried out at potentials of -1.5 V to +1.5 V with a deposition time of 50 seconds and scan rates of 0.05, 0.10, 0.15, 0.20, and 0.25 V/s [13, 10].

RESULTS AND DISCUSSION

Physical characterization of nano titanium dioxide

XRD (X-ray diffraction) analysis is a nondestructive analysis technique used to determine changes in basal spacing in nano titanium dioxide. It is used to determine the crystallinity of a material by describing the relationship between intensity and angle 2θ [20].

The working principle of XRD is when a compound is composed of atoms that form a plane, and light particles come in at a certain angle, resulting in a pattern of reflection or refraction. The pattern of diffraction formed is qualitative analysis data used to distinguish between one compound and another [21]. The following is a graph of the results of the XRD (X-ray diffraction) analysis of nano titanium dioxide:

Based on Figure 1, it can be seen that the graph of pure nano titanium dioxide analysis results is in the anatase phase, as evidenced by the average crystal size of nano titanium dioxide, which is 0.204835549 nm. The figure shows one of the highest peaks of the four known peaks, indicating that the peak is the peak of the formation of titanium dioxide crystallinity with the highest intensity. Judging from the results of the ISSN 1907-1744 (Print) ISSN 2460-1500 (Online)

formation of the XRD diffraction pattern, it means that nano titanium dioxide has a very good crystal value. It is characterized by higher diffraction peaks and narrower angles, which means that the size of nanoparticles increases [22]. The crystal size of nano titanium dioxide can be determined based on the Debye-Sherrer equation as follows:



Figure 1. XRD (X-Ray Diffraction) diffractogram graph of pure nano titanium dioxide

Table 1. Crystal size calculated from the results
of XRD (X-Ray Diffraction) analysis by the
Debye-Sherrer method

Position [°2Th.]	FWHM Left [°2Th.]	Crystal size (nm)
25.2272	0.1673	48.6597
31.6595	0.1004	82.2454
47.8872	0.4684	18.5574
53.7377	0.8029	11.0919
54.9365	0.8029	11.1517
Average c	rystal size	34.3412

Note: FWHM = Full-Width At Half Maximum

XRD (X-ray diffraction) testing aims to determine nano titanium dioxide's structure and intensity size. According to [23]pure nano titanium dioxide analysis results are in the anatase phase, as evidenced by the crystal size and average crystal size of nano titanium dioxide, which can be seen in Table 1. The smaller crystal size can cause a larger surface area of nano titanium dioxide, which helps increase the photocatalyst activity of nano titanium dioxide materials [22].

Chemical characterization of nano titanium dioxide

FTIR (Fourier Transform Infrared) analysis is an organic molecular analysis technique with an infrared range of 4000 cm⁻¹ - 400 cm⁻¹. FTIR (Fourier Transform Infrared) can be used quantitatively because the energy absorbed at a specific wavelength is directly proportional to the amount of kinetic energy associated, so the higher the analyte concentration, the more energy is absorbed [24]. J. Pijar MIPA, Vol. 18 No. 6, November 2023: 986-993 DOI: 10.29303/jpm.v18i6.5844

The working principle of FTIR (Fourier Transform Infrared) is the interaction of energy in the form of infrared rays with material in the form of complex compounds that can cause molecular vibrations. The vibration occurs because the infrared light is not strong enough to cause electron excitation of the tethered molecule. The amount of vibrational energy of each atom or molecule is different depending on the atom and the strength of the connecting bond so that different frequencies can be produced [21]. The following is a graph of the results of FTIR (Fourier Transform Infrared) testing of nano titanium dioxide:



Figure 2. Spectrum graph of FTIR (Fourier Transform Infrared) test results of nano titanium dioxide

Figure 2 is the spectrum of FTIR (Fourier Transform Infrared) testing results of nano titanium dioxide. This method is used to identify the identification results of functional groups and qualitative absorption intensity on nano titanium dioxide. Based on the results of the FTIR test, the absorption of infrared spectra on nano titanium dioxide samples can be seen. FTIR nano titanium dioxide is characterized by the interval distance of wave numbers from 500 cm⁻¹ to 4000 cm⁻¹. The results of FTIR characterization of nano titanium dioxide are Ti-O-Ti groups at a wave number interval of 505 cm⁻ ¹ to 600 cm⁻¹ with a stretching vibrational mode. Ti-O is in the wave number interval 610 cm⁻¹ to 1000 cm⁻¹, which has a stretching vibrational mode. The bending vibrational mode is at the interval number 1700 cm⁻¹ to 3600 cm⁻¹ for the H-O-H group, while at the wave number interval 100 cm⁻¹ to 1500 cm⁻¹, the H-C-H group does not have stretching or bending vibrational modes [25].

According to [23] pure nano titanium dioxide are at peaks of 463.88 and 728.77 cm⁻¹ in the range of 400-800 cm⁻¹ which is an anatase lattice. The broad absorption band between 450 and 800 cm⁻¹ is ascribed to the absorption of Ti-O-Ti vibrations that have an association with nano titanium dioxide. According to the standard spectrum of nano titanium dioxide, the peak is at 463.88 cm⁻¹ and should be attributed to the vibration of the Ti-O bond in the anatase lattice of TiO₂, indicating that the organic ligand is completely removed after annealing at 450°C [23].

Determination of the best electrode composition

Determination of the best electrode composition was carried out with a test method in the form of cyclic voltammetry. The solution tested was 10 mL of 20 ppm chloramphenicol solution in 2000 ppm KCl solution and 5 mL of phosphate buffer solution.

Each electrode was inserted into a voltammetric cell filled with an analyte solution in this test. Tests were conducted at potential limits from -1.5 V to +1.5 V, a deposition time of 10 seconds, and a scan rate of 0.05 V/s. The nano titanium dioxide-modified carbon paste electrode served as the working electrode, while the reference electrode used was Ag/AgCl, and the auxiliary electrode was the Pt electrode.

The best electrode composition test was carried out by measuring the activated nano titanium dioxide modified carbon paste electrode with the composition of carbon: nano titanium dioxide: paraffin oil is 3:2:5, 3:3:4, 3:4:3, and 3:5:2 (w/w). Each electrode was analyzed, and the following results were obtained:



Figure 3. Voltammogram of the highest current peak of the best nano titanium dioxide modified carbon paste electrode composition

The peak currents obtained from all electrode compositions are as follows:

Table 2. Ipc value of the voltammogram determination of the best nano titanium dioxide modified carbon paste electrode

Electrode composition ratio (w/w)	Ipc (A)
5:5	4.74 x 10 ⁻⁵
3:2:5	3.68 x 10 ⁻⁴
3:3:4	5.78 x 10 ⁻⁴
3:4:3	9.04 x 10 ⁻⁴
3:5:2	1.11 x 10 ⁻⁴

Based on Table 2, the peak current (Ipc) of the voltammogram of the best-activated nano titanium dioxide modified carbon paste electrode J. Pijar MIPA, Vol. 18 No. 6, November 2023: 986-993 DOI: 10.29303/jpm.v18i6.5844

with the composition of carbon: nano titanium dioxide: paraffin oil is a ratio of 3:5:2 (w/w). The highest peak current is obtained because it has a high reduction and oxidation peak current [26, 20]. In addition, the 3:5:2 (w/w) ratio has a large composition of nano titanium dioxide to produce a maximum peak current response. The more nano titanium dioxide, the higher the level of analyte absorption because nano titanium dioxide has a large surface area, which can increase the interaction of EPKN with analytes, increasing the intensity of the resulting current [26, 20]. The electrode composition with a ratio of 3:5:2 (w/w) is said to be the best based on less paraffin oil than carbon powder and nano titanium dioxide. More paraffin oil in the composition can inhibit electron transfer between the analyte ions and the electrode, affecting t voltammogram results [27].

Determination of optimum pH

The best nano titanium dioxide-modified carbon paste electrode is used to determine the optimum pH, which can affect the ability and sensitivity of the electrode in testing the analyte solution. Certain optimum pH conditions can make many chloramphenicol ions in the form of free ions so that they can cause these ions to be trapped on the electrode surface and form a chloramphenicol-nano titanium dioxide complex; the resulting current increases.

Determination of optimum pH was carried out using the best electrode composition, namely the ratio of 3:5:2 (w/w). The test was conducted with 10 mL of 20 ppm chloramphenicol solution, 2000 ppm KCl solution, and 5 mL of phosphate buffer (pH 5, 6, 7, 8). The potential is -1.5 V to +1.5 V with a deposition time of 10 seconds and a scan rate of 0.05 V/s. The following is a voltammogram of the highest current peak of the optimum pH:



Figure 4. Voltammogram of the highest current peak of optimum pH

In general, pH is one of the variables that significantly affects the voltammogram's current and shape, as shown in Figure 4, with different pH values, namely pH 5, 6, 7, and 8. The increasing pH value is known, the peak current generated increases, and the optimal current is at pH 8. With the increasing pH value, the size of the cathodic peak will decrease, and the molecules become more easily oxidized so that the wave will shift linearly towards a less positive value [20]. The peak current of the voltammogram determining the optimum pH can be seen in Table 3:

Table 3. Ipc values of the voltam	mograms of th	ıe
optimum pH determir	ation	

рН	Ipc (A)
5	-1.31 x 10 ⁻³
6	-1.48 x 10 ⁻³
7	-1.64 x 10 ⁻³
8	-1.72 x 10 ⁻³

Based on Table 3, it is known that the highest current peak is testing the addition of buffer 8, at that pH chloramphenicol ions in the analyte solution are present in the form of free ions, and then the free ions form complexes with nano titanium dioxide so that the peak current produced is high [13].

Determination of chloramphenicol concentration in vaname shrimp pond water samples by cyclic voltammetry method

Testing of vaname shrimp pond water samples was carried out with chloramphenicol standards of 1, 5, 10, 15, and 20 ppm with a deposition time of 50 seconds, a scan rate of 0.25 V/s, and a potential of -1.5 V to +1.5 V by cyclic voltammetry method. The voltammetry cell was filled with a mixture of standard solutions of 10 mL, each with concentrations of 1, 5, 10, 15, and 20 ppm, added as much as 10 mL of KCl solution with a concentration of 50-100 times the concentration of the standard solution, and added as much as 5 mL of phosphate buffer solution pH 8. The measurement results of the voltammetry method were processed using software in the form of Origin Pro 2018, shown in the following figure:



Figure 5. Voltammogram of the highest current peak of chloramphenicol standard solution with pH 8 buffer solution

Based on Figure 5, the peak current value of each concentration solution analyzed can be known, so it can be used to determine the standard curve of chloramphenicol shown in the following figure:



Figure 6. Standard curve of chloramphenicol cyclic voltammetry method

Linearity can be determined based on the calibration curve that shows the relationship between the response and the concentration of the standard solution. The x-axis is the concentration of chloramphenicol, while the y-axis is the test response or peak current produced. Based on Figure 6 is a graph obtained from the determination of linearity using the equation y = 0.0000303465x + 0.000370882 with r = 0.99861. The linearity relationship can be known based on linear analysis's correlation coefficient (r) value. A linear relationship is a relationship in which the value of r is +1 or -1, depending on the direction of the line [21, 20].

The correlation coefficient (r) value results indicate that this test method has an excellent linear relationship to the testing of chloramphenicol in vaname shrimp pond water using nano titanium dioxide-modified working electrodes because the r value obtained is close to 1 [21]. The value of the correlation coefficient (r) shows the relationship between the two data, showing that the resulting graph is directly proportional. In other words, the greater the concentration of the solution, the more the value of the current increases, while the coefficient of determination shows the diversity of the variable v on the graph [21, 20]. The concentration of chloramphenicol in vaname shrimp pond water samples by cyclic voltammetry method obtained can be seen from the following table:

Table 4. Ipc values of voltammograms of chloramphenicol concentration determination of vaname shrimp pond water samples

Sample	Ipc (A)	Concentration (ppm)
А	5.64 x 10 ⁻⁴	6.37
В	6.52 x 10 ⁻⁴	9.27
С	5.17 x 10 ⁻⁴	4.81
Ave	erage	6.81

Based on the Indonesian Minister of Health Regulation Number 33 in 2012 on Food Additives (BTP), chloramphenicol is a food additive prohibited in Indonesia. According to the Regulation of the Minister of Marine Affairs and Fisheries of the Republic of Indonesia Number 39/PERMEN-KP/2015 concerning Control of Residues of Fish Drugs, Chemicals, and Contaminants in Consumption Fish Farming Activities, and Regulation of the Minister of Marine Affairs and Fisheries of the Republic of 37/PERMEN-KP/2019 Indonesia Number concerning Residue Control in Consumption Fish Farming Activities, it is stated that the chloramphenicol content in fish and shrimp has a BMR (Maximum Residue Limit) of 0 µg/kg, while the BMKL (Minimum Laboratory Performance Limit) is 0.3 µg/kg. In addition, referring to SNI 2729:2013, which states fresh fish's quality and safety requirements, chloramphenicol should not be present in cultured fish.

Table 4 shows that the carbon paste electrode modified nano titanium dioxide can detect chloramphenicol levels in vaname shrimp pond water samples by cyclic voltammetry. The analytical results obtained were sample A at 6.37 ppm, sample B at 9.27 ppm, and sample C at 4.81 ppm. average The concentration of chloramphenicol detected from three different pond water samples in Wangen Village, Glagah District, Lamongan Regency is 6.81 ppm. The concentration of chloramphenicol detected can be said to exceed the threshold of SNI 2729:2013.

This is similar to [28] who analyzed chloramphenicol levels in vaname shrimp farming ponds with concentrations that exceeded the SNI 2729: 2013 threshold of 0.03 µg/kg, 0.062 µg/kg, and 0.041 µg/kg. In addition, [29] showed the results of chloramphenicol residue research that exceeded the SNI 2729 threshold: 2013 that of the six samples of vaname shrimp (cultured in the ponds of Sumberejo Village, Ambulu District, Jember Regency) contained chloramphenicol residues with chloramphenicol residue levels contained from the six samples were 0.14 ppb contained in samples 1K (partial harvest one small), 1B (partial harvest one large), 3K (partial harvest two small), 3B (partial harvest two large), and 0.12 ppb in samples 2K (partial harvest two small) and 2B (partial harvest two large).

Based on the results of this study and previous studies related to chloramphenicol levels in vaname shrimp pond water. Indonesia still needs to monitor the concentration and residual chloramphenicol in vaname shrimp pond water because it can cause several health problems. When exposed to humans, it can cause aplastic anemia, bone marrow suppression, stomach and J. Pijar MIPA, Vol. 18 No. 6, November 2023: 986-993 DOI: 10.29303/jpm.v18i6.5844

intestinal disorders, and optical and peripheral neuropathy [8].

CONCLUSION

Based on the results of the research that has been done, it can be seen that the nano titanium dioxide modified carbon paste electrode is an efficient and capable method for detecting the presence of chloramphenicol compounds in vaname shrimp pond water and can provide a good response. Evidenced by the value of the correlation coefficient (r) obtained close to 1 (one), namely r = 0.99861, and the concentration of chloramphenicol found in vaname shrimp pond water samples, respectively, are 6.37, 9.27, and 4.81 ppm with an average concentration of chloramphenicol in the sample is 6.81 ppm.

REFERENCES

- Anugrah, A., & Alfarizi, A. (2021). Literature Review Potensi dan Pengelolaan Sumber Daya Perikanan Laut di Indonesia. *Jurnal Sains Edukatika Indonesia (JSEI)*, 3(2), 31-36.
- Setiawan, A. (2022). Keanekaragaman Hayati Indonesia: Masalah dan Upaya Konservasinya. *Indonesian Journal of Conservation*, 11(1), 13-21. doi:10.15294/ijc.v11i1.34532
- [3] Febrianti, A., Miftachurrohmah, A., Jannah, A., Muliawan, Y., & Sabil A. A. (2019). Water Quality Controller for Cultivation of Vannamei Shrimp (*Litopenaeus vannamei*) in Wedoro Village, Glagah District, Lamongan Regency. *Journal of Innovation and Applied Technology*, 5(1), 848 - 854.
- [4] Sa'adah, W. (2018). Analisa Kelayakan Usaha Budidaya Udang Vannamei di Desa Dukuh Tunggal, Kecamatan Glagah, Kabupaten Lamongan. Buletin Ilmiah "MARINA" Sosial Ekonomi Kelautan dan Perikanan, 4(1), 33 -37. doi:10.15578/marina.v4i1.7327
- [5] Asfani, D., Hernanda, I., Negara, I., Suwadi, Affandi, Kuswidiastuti, D., Suryani, T. (2023). Alat Pemberi Pakan Udang Otomatis Portabel Berbasis Panel Surya guna Membantu Proses Budidaya Tambak Udang di Desa Tambak Ploso Kabupaten Lamongan. *Sewagati*, 7(3), 353-360. doi:10.12962/j26139960.v7i3.497
- [6] Abudi, C., Koniyo, Y., Suherman, S., & Lamadi, A. (2023). Identifikasi Infeksi WSSV (White Spot Syndrome Virus) pada Udang Vaname (Litopenaeus vannamei) dengan Metode PCR (Polimerace Chain Reaction) di Kecamatan Wanggarasi. Jurnal Ilmiah Multidisiplin, 2(1), 265-273.
- [7] Visakhadevi, J., Hidayati, N., Fitrani, M., & Andriyono, S. (2023). Aplikasi Metode ELISA dalam Deteksi Residu Antibiotik

Kloramfenikol pada Produk Olahan Perikanan. *PoluSea*, 1(1), 1-9.

- [8] Sari, J., & Hafludin. (2023). Analisis Kadar Residu Antibiotik Kloramfenikol pada Udang Vannamei (*Litopenaeus vannamei*) di Kabupaten Bangkalan dengan Metode ELISA (Enzym Linked Immunosorbent Assay). *Juvenil*, 4(2), 84-89. doi:10.21107/juvenil.v4i2.18075
- [9] Habibah, Z., & Setiarso, P. (2016). Pembuatan Elektroda Pasta Karbon Termodifikasi Bentonit untuk Analisis Ni(II) pada Kerang Darah (*Anadara granosa*) secara Voltametri Siklik. UNESA Journal of Chemistry, 5(2), 94-99.
- [10] Rahayu, R. (2020). Analisis Kadar Klorin (Cl) dalam Beras dengan Metode Voltametri Siklik Menggunakan Elektroda Platina (Pt). *Laporan Tugas Akhir*. Yogyakarta: Universitas Islam Indonesia.
- [11] Cahyotomo, A., Panglipur, H., Tirta, A., & Madiabu, H. (2022). Deteksi Metil Paraben secara Voltametri Menggunakan Elektroda Pasta Karbon. *Warta Akab*, 46(1), 16-20. doi:10.55075/wa.v46i1.79
- [12] Suprasetyo, A., & Setiarso, P. (2016). Pembuatan Elektroda Pasta Karbon Termodifikasi Zeolit untuk Analisis Fenol Cyclic Stripping Voltammetry. UNESA Journal of Chemistry, 5(3), 86-93.
- [13] Inggriani, F., & Setiarso, P. (2021). Pembuatan Elektroda Pasta Karbon untuk Analisis Parasetamol secara Differential Pulse Voltametri. *Indonesian Chemistry and Application Journal*, 4(2), 27-31. doi:10.26740/icaj.v4n2.p27-31
- [14] Puspita, F., & Noviandri, I. (2021). Optimasi Pembuatan Elektroda Pasta Karbon Termodifikasi Poli(Metil Jingga) untuk Penentuan Bisphenol A secara Voltametri. Warta Akab, 45(1), 14-20.
- [15] Wei, Q., Wu, L., Zhu, M., Wang, Z., Huang, Z., & Wang, M. (2023). Porous Nitrogen-Doped Reduced Graphene Oxide-Supported CuO@Cu₂O Hybrid Electrodes for Highly Sensitive Enzyme-Free Glucose Biosensor. *iScience*, 26(3), 1-17. doi:https://doi.org/10.1016/j.isci.2023.106155
- [16] Wulandari. (2023). Uji Aktivasi Antioksidan Senyawa Fukosantin dari Cyclotella striata Menggunakan Metode Voltametri Siklik. Skripsi. Bandar Lampung: Universitas Lampung.
- [17] Faria, L., Lisboa, T., Alves, G., Matos, M., Munoz, R., & Matos, R. (2021). Adsortive Stripping Voltammetric Determination of Chloramphenicol Residues in Milk Samples

J. Pijar MIPA, Vol. 18 No. 6, November 2023: 986-993 DOI: 10.29303/jpm.v18i6.5844

using Reduces Graphene Oxide Sensor. Analytical Methods, 13(47), 5711-5718. doi:10.1039/D1AY01756J

- [18] Juliana, M., & Yulian, M. (2020). Identifikasi Kloramfenikol pada Udang Vaname (*Litopenaeus vannamei*) Menggunakan Liquid Chromatography (HPLC). AMINA, 2(1), 13-18. doi:10.22373/amina.v2i1.493
- [19] Tumanduk, R., Massi, M., Agus, R., & Hamid, F. (2023). Analisis Residu Amoksilin pada Hepar dan Ventrikulus Ayam Petelur di Pasar Tradisional Makassar. Jurnal Ilmu Alam dan Lingkungan, 14(2), 20-28. doi:10.20956/jal.v14i2.27900
- [20] Triana, S. (2020). Elektroda Pasta Karbon Termodifikasi TiO₂ Nano untuk Deteksi Bisfenol-A (BPA) dalam Air Mineral secara Voltametri Siklik. *Laporan Tugas Akhir.* Yogyakarta: Universitas Islam Indonesia.
- [21] Ariani, F. (2020). Pengembangan Elektroda Kerja Berbasis EPK/TiO₂ untuk Monitoring Bisphenol-B pada Plastik secara Voltametri Siklik. Skripsi. Yogyakarta: Universitas Islam Indonesia.
- [22] Syuhada, N. (2023). Fabrikasi dan Uji Luas Permukaan Material Microsphere-Nanorod Titanium Dioksida. *Spin*, 5(1), 146-155. doi:10.20414/spin.v5i1.7157
- [23] Abdallah, H., Adarosy, M. H., Hegazy, H. S., & Abdelhameed, R. E. (2022). Potential of Green Synthesized Titanium Dioxide Nanoparticles for Enhancing Seedling Emergence, Vigor and Tolerance Indices and DPPH Free Radical Scavenging in Two Varieties of Soybean under Salinity Stress. BMC Plant Biology, 22(1), 1-18. doi:10.1186/s12870-022-03945-7
- [24] Nurfirzatulloh, I., Suherti, I., Insani, M., Shafira, R., & Abriyani, E. (2023). Literature Review Article: Identifikasi Gugus Fungsi Tanin pada Beberapa Tumbuhan dengan Instrumen FTIR. Jurnal Ilmiah Wahana Pendidikan, 9(4), 201-209. doi:10.5281/zenodo.7678425
- [25] Kundiman, N., Kurnia, Indrayana, I., & Sadjab, B. (2023). Pengaruh Variasi Suhu Kalsinasi terhadap Parameter Mikrostruktur (Struktur Kristal dan Gugus Fungsi) Nanopartikel TiO₂ sebagai Kandidat Material Fotokatalitik. *Journal Online of Physics*, 8(3), 14-20. doi:10.22437/jop.v8i3.26814
- [26] Astuti, A., & Ningsi, S. (2017). Sintesis dan Karakterisasi Nanopartikel Titanium Dioksida (TiO₂) Menggunakan Metode Sonokimia. *Jurnal Ilmu Fisika Universitas Andalas*, 9(1), 26-32. doi:10.25077/jif.9.1.26-32.2017

- [27] Wahyuni, W., Darusman, L., & Herlina, D. (2017). Fabrication and Analitical Performance Evaluation of Carbon Paste Electrode using Cyclic Voltammetry. *Analytical and Enviromental Chemistry*, 2(2), 12-25.
- [28] Suseno, H., Hudiyono, S., & Muslim, M. (2016). Elimination of Chloramphenicol by Tiger Shrimp (*Panaeus monodon*) and White Shrimp (*Litopenaeus vannamei*). *HAYATI Journal of Bioscience*, 23(3), 117-120. doi:10.1016/j.hjb.2016.07.001
- [29] Virgianti, S., Moelyaningrum, A., & Ningrum, P. (2022). Kandungan Residu Kloramfenikol pada Udang Putih (*Litopenaeus vannamei*). *Buletin Keslingmas*, 41(4), 149-155. doi:10.31983/keslingmas.v41i4.9332
- [30] Badan Pengawas Obat dan Makanan. (2023). Bahan Tambahan yang Dilarang Digunakan dalam Produk Makanan. Retrieved Juni 20, 2023, from https://standarpangan.pom.go.id/berita/bahantambahan-yang-dilarang-digunakan-dalamproduk-pangan
- [31] Peraturan Menteri Kesehatan RI. (2012). Peraturan Menteri Kesehatan Republik Indonesia Nomor 33 Tahun 2012 tentang Bahan Ta,bahan Pangan (BTP). Jakarta: Kementerian Kesehatan Republik Indonesia.
- [32] Badan Standarisasi Nasional. (2013). Persyaratan Mutu dan Keamanan Ikan Segar. Jakarta: Badan Standarisasi Nasional.
- [33] Peraturan Menteri Kelautan dan Perikanan RI. (2015). Peraturan Menteri Kelautan dan Perikanan RI Nomor 39 Tahun 2015 tentang Pengendalian Residu Obat Ikan, Bahan Kimia, dan Kontaminan pada Kegiatan Pembudidayaan Ikan Konsumsi. Jakarta: Kementerian Kelautan dan Perikanan Republik Indonesia.
- [34] Peraturan Menteri Kelautan dan Perikanan RI. (2019). Peraturan Menteri Kelautan dan Perikanan RI Nomor 37 Tahun 2019 tentang Pengendalian Residu pada Kegiatan Pembudidayaan Ikan Konsumsi. Jakarta: Kementerian Kelautan dan Perikanan Republik Indonesia.