

Determination of Hydrazine in Various Water Samples by Square Wave Voltammetry with Zinc-Layered Hydroxide-3(4-methoxyphenyl) Propionate Nanocomposite Modified Glassy Carbon Electrode

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The application of zinc-layered hydroxide-3(4-methoxyphenyl) propionate (ZLH-MPP) nanocomposite as a suitable mediator in a voltammetric sensor for the determination of hydrazine by square wave voltammetry (SWV) were evaluated. The ZLH-MPP modified glassy carbon electrode exhibited a good sensitivity in 0.7 M Na₂SO₄, pH 10.0 and using square wave parameter of 120 Hz frequency, 25 mV pulse size and 3 mV step size. The SWV response showed a linear dependence of the peak current to the concentration in the range of 1.0×10^{-6} – 1.0×10^{-4} M hydrazine with limit of detection 6.7×10^{-7} M and correlation coefficient of 0.9966. The presence of 200 fold of metal ions and 500 fold organic substances in excess to the concentration of hydrazine did not interfere. Electrochemical impedance spectroscopy showed that the charge transfer at the electrode-solution interface was favourable. The proposed electrode was applied for the recovery of the real samples studies by using standard addition method.

Keywords: glassy carbon electrode, hydrazin, hydroxide-3(4-methoxyphenyl) propionate, square wave voltammetry

1. INTRODUCTION

Hydrazine, with the formula of N₂H₄, is an inorganic compound and has a molecular weight of 32.05 g/mol. It is a colourless, oily, flammable liquid which is miscible with water and has an

ammonia-like odor. Hydrazine and its derivative has many uses such as fuel for certain rocket and spacecraft, as an oxygen scavenger in boiler feed water in order to inhibit corrosion, insecticides, photographic chemicals, explosives blowing agents for plastics and as a halogen removal from wastewater [1,2].

People may be exposed to hydrazine in the workplace, cigarette, and from waste disposal into the environment and improper handling. Nonetheless, hydrazine rapidly degrades in the environment and is rarely encountered for. However, because of its toxicity level which is $0.002 \mu\text{g}/\text{m}^3$, the Environmental Protection Agency (EPA) has classified hydrazine and its derivative as environmental pollutants [3]. There are several symptoms of acute exposure to high levels of hydrazine including irritation of the eyes, nose, and throat, temporary blindness, dizziness, headache and nausea. More severe exposure will lead to damage to the brain, skin, liver and kidneys. Hence, based on these effects serious attention has been focused on the quantitative determination of hydrazine [4,5].

Several methods on the determination of hydrazine have been proposed. They are spectrophotometry [6], amperometry [7,8], potentiometry [9], chemiluminescence [10] and flow injection analysis [11]. Among these electrochemical methods are more sensitive, economical, effective and rapid [4,12]. But, the irreversible oxidation of hydrazine requires large overpotential at glassy carbon electrodes (GCE). This, however, may be minimized through various chemical modifications of the electrodes. The use of various of redox mediators like alizarin [13], acetylferrocene [14], quinizarine [15], *o*-aminophenol [16], 1,1'-ferrocenedicarboxylic acid [1] and pyrogallol red [17] have been used for this purpose.

In this work, the determination of hydrazine have been studied using zinc-layered hydroxide-3(4-methoxyphenyl) propionate (ZLH-MPP) nanocomposite modified GCE.

2. EXPERIMENTAL

2.1. Reagents and chemicals

Hydrazine hydrate, sodium sulphate, 0.25% nafion, sodium hydroxide and hydrochloric acid (Merck, Germany) were used as received. All analytical grade supporting electrolytes such as sodium sulphate, sodium chloride, potassium nitrate and sodium nitrate were supplied by Sigma-Aldrich, USA. The complex of ZLH-MPP was synthesized and purified as previously reported [18]. All solutions were prepared with doubly distilled deionized water from Barnstead EasyPure LF, USA. The pH was adjusted by drop wise addition of of hydrochloric acid and sodium hydroxide.

2.2. Apparatus

A GCE (id = 3mm) was purchased from Bioanalytical Instrument (USA). SWV and the electrochemical impedance spectroscopy (EIS) were performed with a Potentiostat Gamry Series-G 750 (USA). All the experiments were carried out in 0.7 M Na_2SO_4 in a three-electrode electrochemical cell. GCE was used as a working electrode, a platinum wire was used as a counter electrode and

saturated Ag/AgCl act as a reference electrode. The pH of the solution was adjusted using Orion 720A plus meter of Thermo Electron Corp. (USA) with glass electrode Orion 915600 (USA). Prior to any measurement, the solutions were deoxygenated by purging the nitrogen gas (Nissan Oxygen, Malaysia). The morphological surface of the modifier was characterized using Hitachi model SU 8020 UHR field emission scanning electron microscope (FESEM) (Japan). The XRD patterns were carried out using power diffraction Bruker AXS model D8 Advance (Italy), wavelength of 1.5406 Å and the energy-dispersive X-ray spectroscopy (EDX) analysis were recorded using model Hitachi SU 8020 UHR (Japan).

2.3. Synthesis ZLH-MPP nanocomposite

The ZLH nanocomposite was prepared by direct reaction of ZnO with the guest anion of 3(4-methoxyphenyl) propionic acid (MPP) (Fisher Scientific, UK) in an aqueous environment as previously reported [18]. The 0.05 M MPP was prepared by dissolving the required amounts in 50 mL 99.8% methanol. The MPP solutions were mixed with 0.50 g ZnO with 20 mL deionised water. The mixture were stirred for 2 hours. Then, 20 mL of deionised water was added in an oil bath shaker before aging for 24 hours at 70 °C. The slurry was centrifuged, rinsed with deionised water and then dried for 24 hours in oven. The samples were then kept in a bottle for further characterization and use.

2.3. Electrode preparation

The GCE surface was polished by using polish pad and a 0.5 mm alumina slurry (BAS, USA). After rinsing thoroughly with water, the GCE was sonicated in a mixture of water/ethanol to remove any possible retained alumina particles on the electrode surface. A 1.0 mg of ZLH-MPP modifier was dispersed in 1 mL of 0.25 % Nafion and sonicated for 3 minutes. Then, 3 µL of ZLH-MPP suspension was dropped on the surface of a bare GCE and solvent was allowed to evaporate in the air in about 30 minutes. Fig. 1 illustrated the preparation of the modified electrode.

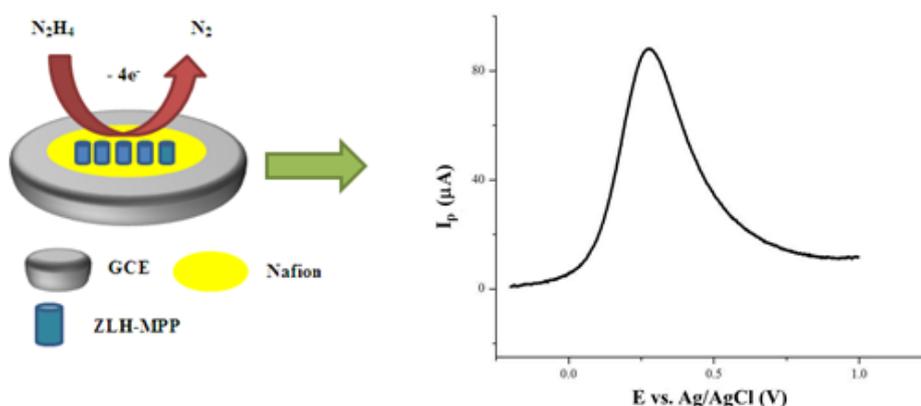


Figure 1. Schematic diagram of the electrode reaction on hydrazine and its anodic peak current.

2.4. Chronocoulometry study

A double potential-step chronocoulometry experiment was carried out on 1.0×10^{-4} M hydrazine in 0.7 M Na_2SO_4 at pH 10.0. The double potentials-step chronocoulometric measurement was applied the initial potential step from -0.89 to 0.35 V vs. Ag/AgCl.

3. RESULTS AND DISCUSSION

3.1. The characterization of ZLH-MPPnanocomposites

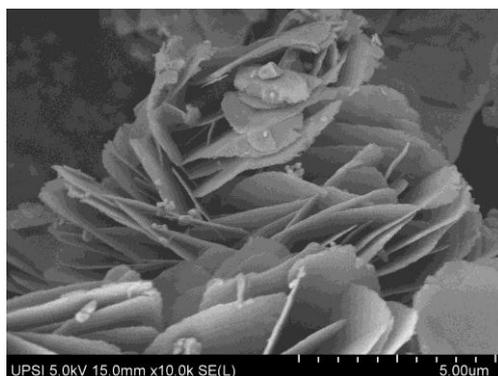


Figure 2. The FESEM micrograph of ZLH-MPP at 10,000X magnification.

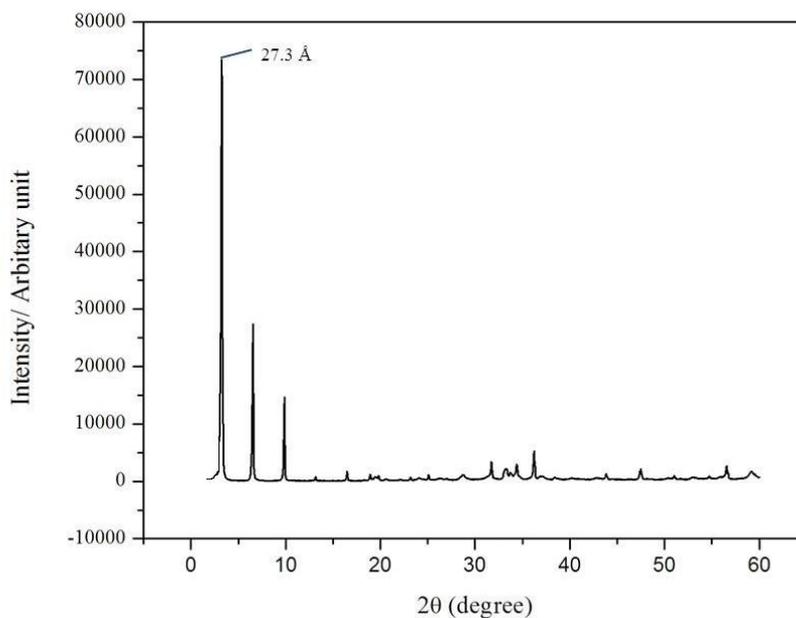


Figure 3. XRD pattern of ZLH-MPP nanocomposite.

Fig. 2 shows the surface morphology of ZLH-MPP nanocomposite indicating a plate-like particle [19] and a monodisperse and flower-like microspheres constructed by the ZnO nanosheets [20]. This is a typical morphology of ZLH and other materials such as Layered Double Hydroxide

(LDH) and its nanocomposite are arranged closely to each other and consists of plate-like surface with a few of granular shapes on it [21, 22].

The XRD pattern of the ZLH-MPP nanocomposites (Fig. 3) indicated intercalation of 0.05 M of MPP within ZLH nanocomposites was exhibited a diffraction pattern with a sharp diffraction peak at low 2θ angle centered with d spacing at 27.3 Å. These sharp peak obtained was due to high crystallinity of material and well ordered stacking layer [23,24]. The existence of small peak of ZnO between 32° to 37° indicated an incomplete reaction of ZnO phase [25]. The EDX spectrum (Fig. 4) shows the ZLH-MPP nanocomposites were comprised of carbon, oxygen and zinc elements with wt % of 34.61 %, 29.70 % and 35.69 %, respectively.

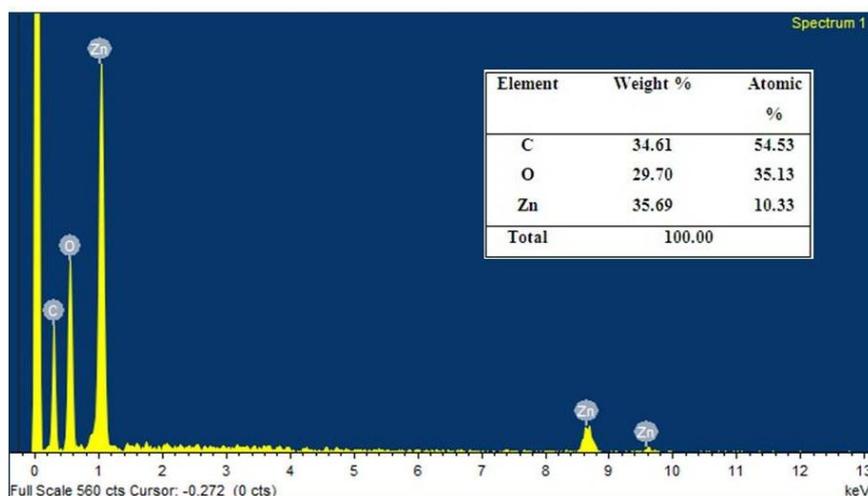
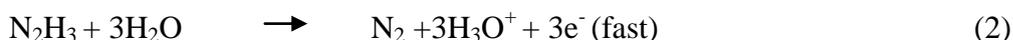
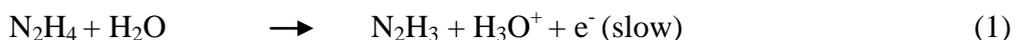


Figure 4. EDX spectrum of ZLH-MPP nanocomposite.

3.1. Electrochemical behavior of hydrazine on ZLH-MPP modified GCE

The electrochemical behavior of 1×10^{-4} M hydrazine in 0.7 M of sodium sulphate (pH = 10) on ZLH-MPP modified GCE was studied by SWV. Fig. 5 shows that the ZLH-MPP modified GCE has its peak current increased significantly. The peak potential of hydrazine is shifted negatively from 0.51 V and 0.32 V indicating a catalytic process. These results clearly indicate that the overpotential oxidation of hydrazine on ZLH-MPP modified GCE is reduced, hence, enhancing the signal of hydrazine.

The following mechanism may be proposed for the oxidation of hydrazine in presence of ZLH-MPP nanocomposites at the surface of GCE;



Overall, the oxidation of hydrazine can be summarized as in the following;



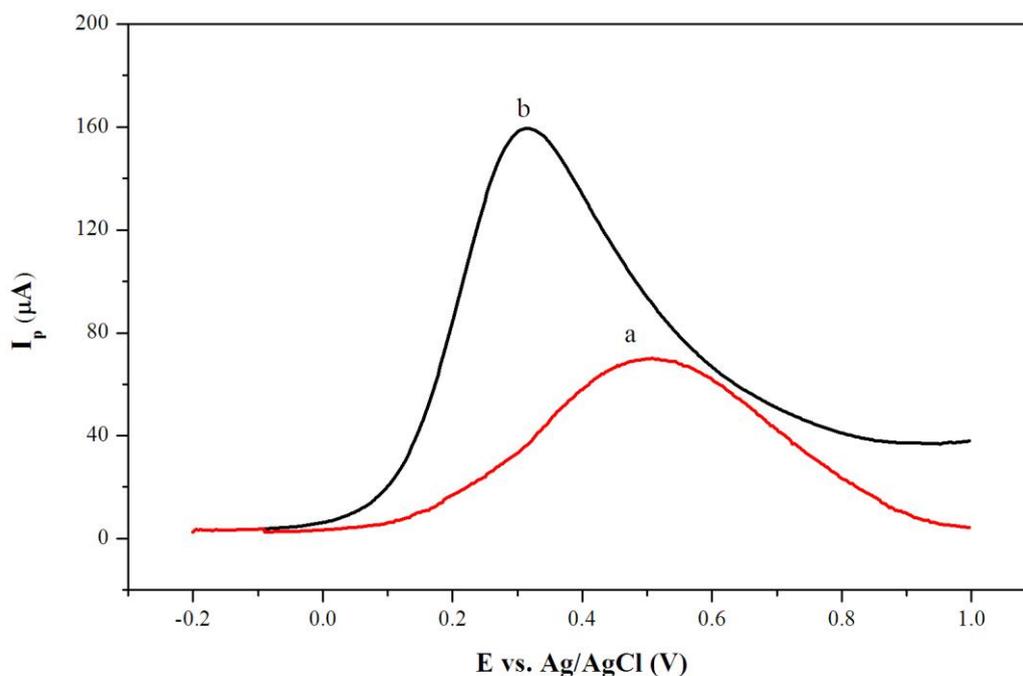


Figure 5. SWV of (a) unmodified GCE and (b) ZLH-MPP modified GCE towards 1.0×10^{-4} M hydrazine in 0.7 M Na_2SO_4 at pH 10.

3.2. Electrochemical impedance spectroscopy (EIS) studies

EIS is a technique in measuring and characterising the interfacial electron transfer resistance of the different electrodes [5,26]. The Nyquist plot of the unmodified GCE and ZLH-MPP modified GCE in 0.7 M sodium sulphate (Fig. 6). The ZLH-MPP modified GCE exhibited the smaller diameter of semicircles indicating lower electron-transfer resistance, R_{ct} . The R_{ct} values for unmodified GCE and ZLH-MPP modified GCE are 4354 ohms and 878.1 ohms, respectively. The straight-line at lower frequency for both electrodes indicated the diffusion process. By fitting the Randles equivalent electrical circuits (Fig. 6 inset) the electron transfer apparent rate constant, k_{app} , for 1.0×10^{-4} M hydrazine on the unmodified GCE and ZLH-MPP modified GCE was calculated according to this equation:

$$k_{app} = RT / F^2 R_{ct} C \quad (4)$$

Where R is the gas constant, T is the temperature, F is a Faraday's constant and C is the concentration of the hydrazine. The k_{app} for unmodified GCE and ZLH-MPP modified GCE are $5.93 \times 10^{-2} \text{ cm s}^{-1}$ and $2.94 \times 10^{-1} \text{ cm s}^{-1}$, respectively. The high value of k_{app} and the low value of R_{ct} of the ZLH-MPP modified GCE indicated the electrode is good to measure hydrazine.

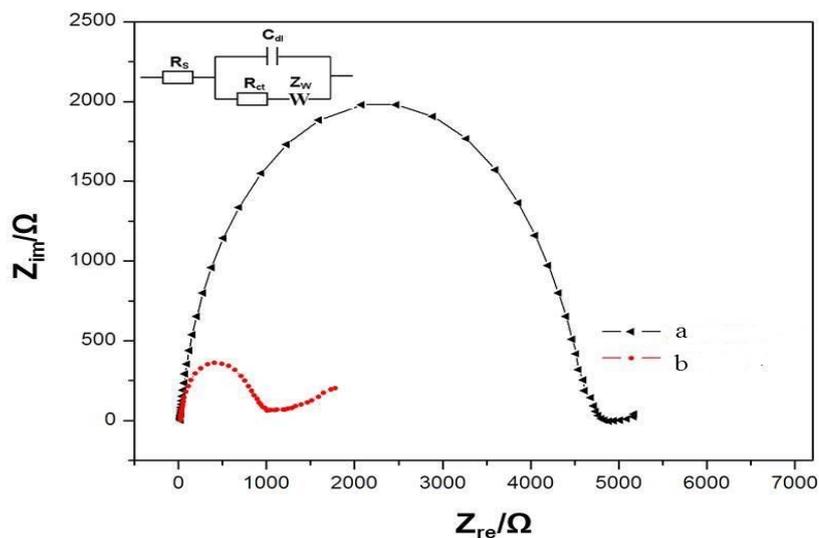


Figure 6. EIS of (a) bare GCE and (b) ZLH-MPP/GCE in 1×10^{-4} M hydrazine in 0.7 M Na_2SO_4 (pH 10).

3.3. Optimization of the experimental conditions

3.3.1. Effect of pH

The effect of pH of the electrolyte towards the oxidation of hydrazine has been investigated over pH range of 5-12. Fig. 7 shows as the pH of the electrolyte increases, the anodic peak current, I_{pa} , of hydrazine also increases until optimum at pH 10. The peak current decreases beyond that i.e. pH 11 and 12. The decrease of the peak currents is due to the protonation or deprotonation of hydrazine [27].

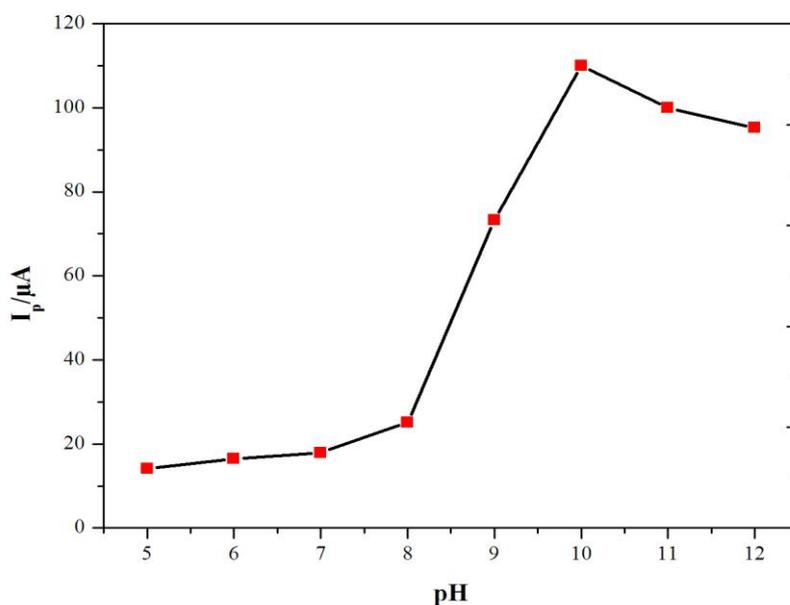


Figure 7. Plot of I_p vs. pH of 1×10^{-4} M of hydrazine in 0.7 M Na_2SO_4 .

3.3.2. Effect of square wave parameter

Cyclic voltammetry is widely used but it is less sensitive than SWV. The square wave signal depends on pH and the electroanalytical parameter [28]. Hence, the optimization of square wave parameters like frequency, step size and pulse size were investigated. The frequency (20-160 Hz) shows a linear relationship. As the frequency increase, the I_{pa} of the hydrazine also gradually increases until it reaches a constant value over 120 Hz as shown in Fig. 8a. Thus, 120 Hz was chosen as an optimum frequency in order to enhance the sensitivity without any changing of peak potential. Then, we have changed the pulse size parameter (20-100 mV) at a fixed frequency, 120 Hz. As we increase the pulse size, the peak current also increases linearly but 25 mV was chosen as the best pulse size as it generated a good resolution. When we varied the step size (1-10 mV), the peak current was essentially the same which increased linearly as the step size increase but then 8 mV was chosen as the optimum step size with a sharper peak.

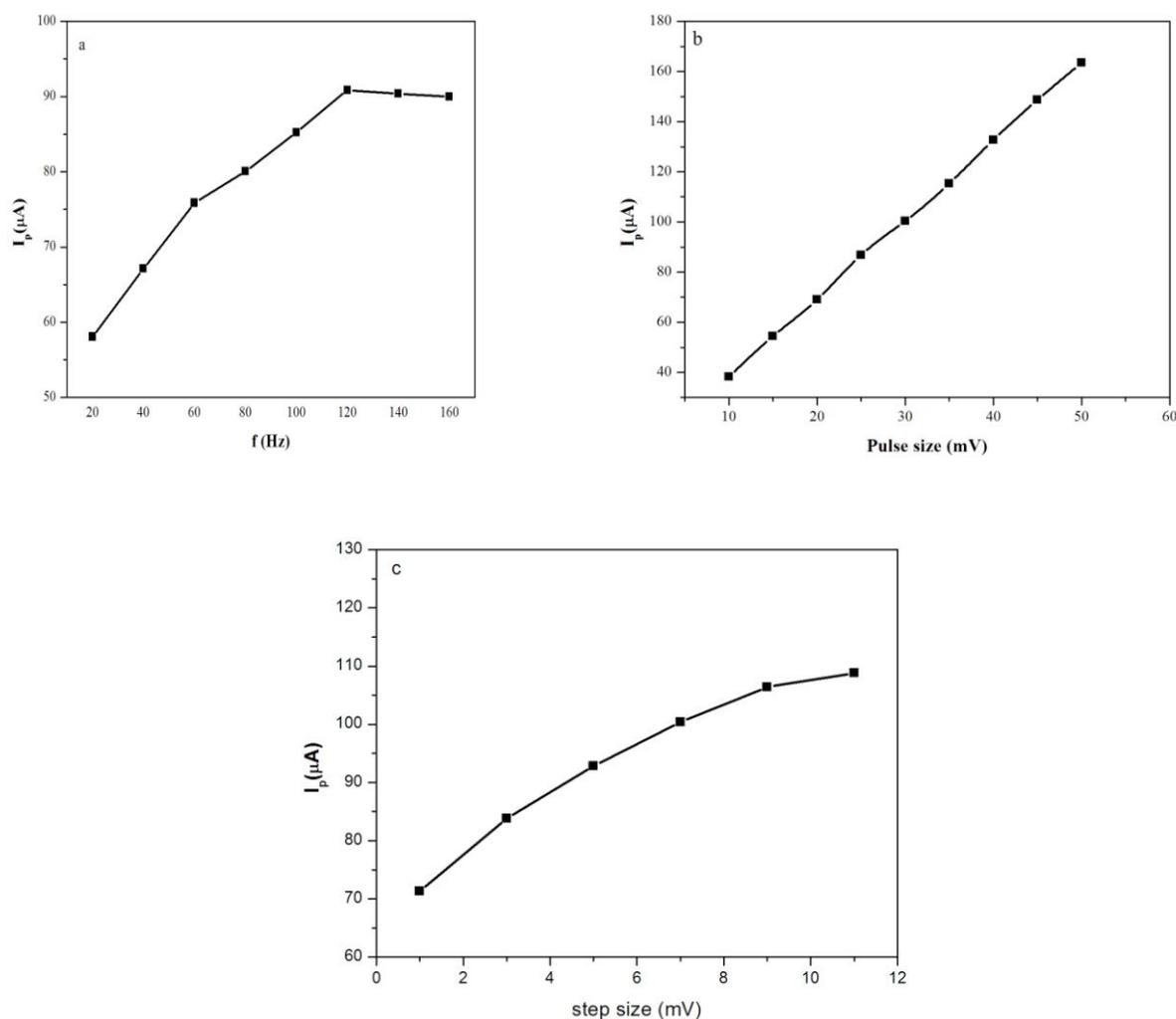


Figure 8. Influence of the square wave parameter (a) frequency (b) pulse size and (c) step size on SWV signal of 1×10^{-4} M hydrazine, pH 10.

3.4. Calibration curve

The proposed electrode was applied in the determination of hydrazine in 0.7 M Na₂SO₄ at pH 10.0 by SWV with frequency of 120 Hz, pulse size of 25 mV and step size of 3 mV. The SWV signal for different concentrations of hydrazine and the relative calibration plots were illustrated in Fig. 9. The result shows that the I_{pa} is linearly increased with the increasing of hydrazine concentration of 1.0 × 10⁻⁶ to 1.0 × 10⁻⁴ M with correlation coefficients, R², 0.9966 and limit of detection of 6.7 × 10⁻⁷ M hydrazine. The limit of detection was assessed by linear regression of the calibration curve by using these equation

$$LOD = 3\sigma/S \tag{5}$$

Where σ is a standard deviation of the response and S is the slope of calibration curve. As can be seen, the calibration curve shows good linearity and the results so obtained compared favorably if not better than many other reported hydrazine detector (Table 1).

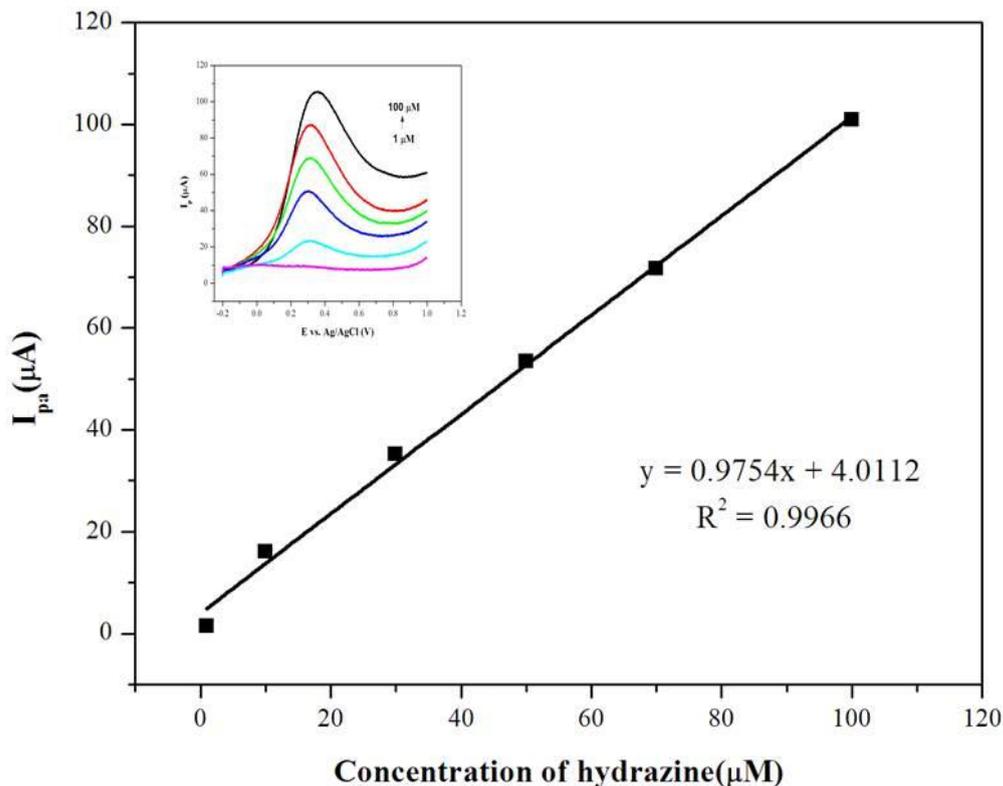


Figure 9. A plot of I_{pa} vs concentration of hydrazine. (inset is SWV of different concentration of hydrazine).

Table 1. Parameters of the modified GCE for determination of hydrazine.

Electrode	Method	Concentration range (M)	Detection limit (M)	Refs.
1,1'-Ferrocenedicarboxylic acid/GCE	CV ^a	$4.0 \times 10^{-5} - 1.0 \times 10^{-3}$	2.6×10^{-5}	[1]
MnHCF/GWCE	Amperometry	$3.3 \times 10^{-5} - 8.2 \times 10^{-3}$	6.7×10^{-6}	[8]
Alizarin/GCE	LSV ^b	$1.0 \times 10^{-5} - 8.0 \times 10^{-4}$	4.6×10^{-6}	[13]
<i>o</i> -aminophenol/GCE	Amperometry	$2.0 \times 10^{-6} - 2.0 \times 10^{-5}$	5.0×10^{-7}	[16]
Ruthenium/GCE	FIA ^c	$1.0 \times 10^{-5} - 1.0 \times 10^{-2}$	8.5×10^{-6}	[29]
CuO nanoparticles/GCE	DPV ^d	$2.5 \times 10^{-8} - 1.7 \times 10^{-3}$	2.0×10^{-5}	[30]
Poly Ni (II) complex/GCE	CV	$1.0 \times 10^{-6} - 1.0 \times 10^{-4}$	4.0×10^{-5}	[31]
A salophen derivatives	CV	$1.0 \times 10^{-5} - 4.0 \times 10^{-4}$	1.0×10^{-6}	[32]
CM/MWCNT/GCE	Amperometry	$2.0 \times 10^{-6} - 4.4 \times 10^{-5}$	1.4×10^{-6}	[33]
Pd nanowires/CILE	SWV ^e	$5.0 \times 10^{-6} - 8.0 \times 10^{-4}$	8.2×10^{-7}	[34]
Nano-Au/Porous-TiO ₂ /GCE	Amperometry	$3.0 \times 10^{-6} - 3.0 \times 10^{-4}$	1.0×10^{-6}	[35]
Ni(II)/BA/MWCNT/PE	Amperometry	$2.5 \times 10^{-6} - 2.0 \times 10^{-4}$	8.0×10^{-7}	[36]
Au/Ti	LSV	$5.0 \times 10^{-4} - 4.0 \times 10^{-3}$	4.2×10^{-5}	[37]
BiHCF/GCE	CV	$7.0 \times 10^{-6} - 1.0 \times 10^{-3}$	3.0×10^{-6}	[38]
ZLH-MPP/GCE	SWV	$1.0 \times 10^{-6} - 1.0 \times 10^{-4}$	6.7×10^{-7}	This work

^a Cyclic voltammetry.

^b Linear sweep voltammetry.

^c Flow injection analysis.

^d Different pulse voltammetry.

^e Square wave voltammetry.

3.5. Chronocoulometry studies

The measured currents were integrated to give a linear Anson plot of charge (Q) vs. the square root of time ($t^{1/2}$), as shown in Figure 10. Then, according to the Cottrell equation, the charge of adsorbed species (Q_{ads}) and diffusion coefficient (D) of ZLH-MPP modified GCE can be calculated from the slope and intercept, respectively [39,40]. The charge of adsorbed species, Q_{ads} is the difference between the intercept of the pair lines and the surface coverage of the electrode, Γ can be calculated using the following equation;

$$Q_{ads} = nFA\Gamma \quad (6)$$

Where, n is the number of electrons transferred, F is Faraday constant (96485 C/equiv), A is area of the electrode surface (cm^2) and Γ is the surface coverage of electrode (mol cm^{-2}). For an area of 0.9425 cm^2 the Q_{ads} was $525.1 \mu\text{C}$ and the surface coverage was $1.45 \times 10^{-9} \text{ mol cm}^{-2}$. The diffusion coefficient, D , of ZLH-MPP modified GCE was $2.85 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$. The value of D was in agreement with the previous study which was $5.9 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$ [16]. This indicates that the developed electrode has a high sensitivity in determination of hydrazine.

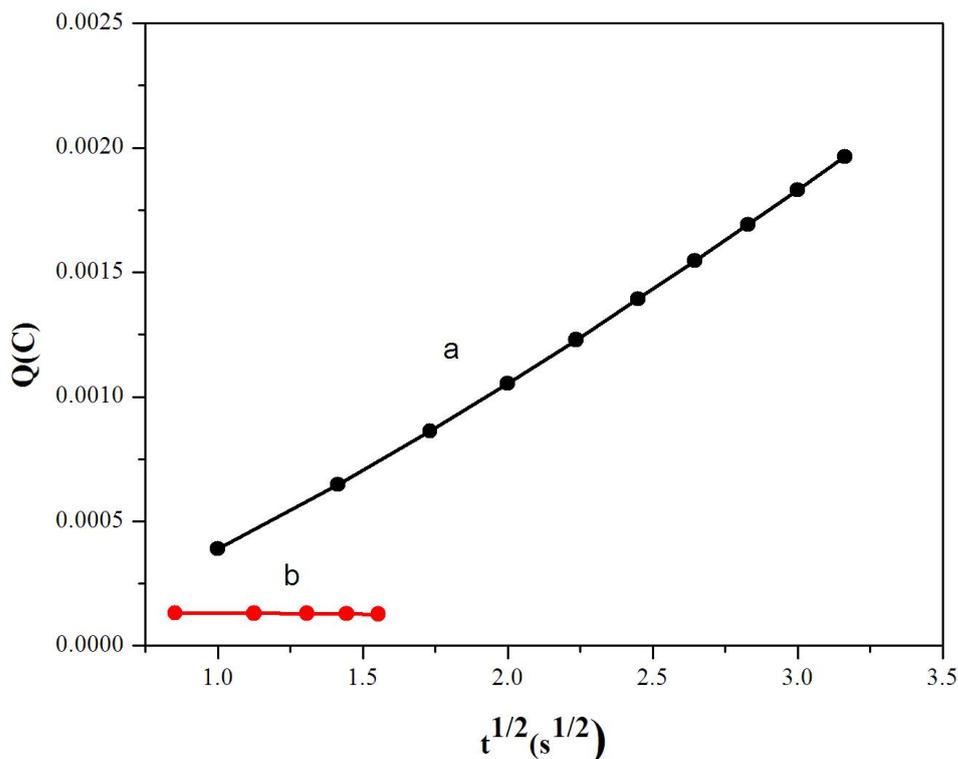


Figure 10. Double potential step chronocoulograms, line a (first potential step) and line b (second potential step) of 1×10^{-4} M hydrazine in 0.7 M Na_2SO_4 (pH 10) at ZLH – MPP/GCE at scanning potential of $-0.89 - 0.35$ V.

3.6. Reproducibility and stability

The reproducibility and stability of the ZLH-MPP/GCE were carried out for 1×10^{-4} M hydrazine in 0.7 M Na_2SO_4 , pH 10.0. The relative standard deviation (RSD) was 4.60 % for ten measurements with the same electrode. After one month storage in 0.7 M Na_2SO_4 , pH 10.0, the current response remained 90% of the original value. The results indicated the excellent reproducibility and storage stability of the proposed modified electrode for detection of hydrazine.

3.7. Interference studies

In order to investigate the influence of various interference substances the proposed electrode was studied of its tolerance limit under the optimum conditions in 1.0×10^{-4} M hydrazine in 0.7 M Na_2SO_4 pH 10. The tolerance limit was defined as the maximum concentration of interferent substances which cause an error of less than 5%. The results indicated that 500 fold excess of glucose, sucrose, fructose and lactose and 200 fold excess of Na^+ , Ba^+ , Ca^{2+} , Cd^{2+} , K^+ , Cl^- , NO_3^- , CH_3COO^- and CO_3^{2-} do not affect the peak current of hydrazine. This clearly proved the the proposed electrode is selective for the determination of hydrazine.

3.8. Recovery of hydrazine in real samples

In order to evaluate the applicability of the proposed electrode in real samples, the standard addition method was applied in the determination of hydrazine in different water samples such as tap water, pond water and river water. As shown in Table 2, the recoveries for the determination of hydrazine were from 94 % to 108 % and the RSD was below 5% (n = 3) which indicates that the presented method could be efficient and reliable for determination of hydrazine in real samples.

Table 2. Determination of hydrazine in different water samples (n=3).

Samples	Hydrazine added (μM)	Hydrazine found (μM)	Recovery (%)	RSD (%)
Pond water	-	-	-	-
	10	9.54	95	2.9
	50	54.00	108	3.8
Tap water	-	-	-	-
	10	9.50	95	1.4
	50	54.00	107	2.3
River water	-	-	-	-
	10	9.60	96	1.5
	50	49.60	99	1.0

4. CONCLUSIONS

This study has demonstrated that the construction and viability of ZLH-MPP/GCE by using zinc-layered hydroxide-3(4-methoxyphenyl) propionate nanocomposite as a mediator in determination of hydrazine by square wave voltammetry. The proposed electrode is sensitive, selective, simple, and low cost and can be applied in the analysis of different water samples.

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