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Voltammetric Determination of Paracetamol with Carbon Paste Electrode Modified with Molecularly Imprinted Electropolymer

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ABSTRACT. Paracetamol is a commom analgesic and antipyretic drug which used for reliefing fever and head ache. The determination of paracetamol dose in pharmaceuticals is very important, becauce an overdose can cause fulminating hepatic necrosis and other toxic effects. Therefore, it is necessary to measure the dose of paracetamol for the patient with precision to avoid harm. Many analytical methodologies have been proposed for determination of paracetamol dose. One of the methods was developed in the past two decades. Generally, electroanalytical approach especially voltammetry method is particularly design for determination of paracetamol dose especially in modifying electrode. This study aims to modified carbon paste electrode with *molecularly imprinted polymer* (MIP). Significant advantages of using MIP are the superior stability, low cost and ease of preparation. The poly (3-aminiophenol) film was prepared by cyclic voltammetry method and 3-aminophenol monomer in supporting electrolyte ($HClO_4$) with and whitout presence of paracetamol molecule. The effect of paracetamol was seen at cyclic voltammogram was founded, where oxidation peak potential of poly (3-aminophenol) shifted to more cathodic potentials from 0.948 to 0.780 V, in presence of paracetamol. The Ipa showed a good linear relationship with concentration in the range 0.01–0.1 mM, and the detection limit was 4,63 μM.

Keywords: Molecularly imprinted polymer, paracetamol, poly(3-aminophenol), electropolimerization and carbon paste electrode.

INTRODUCTION

Paracetamol is an acylated aromatic amide, which was first introduced to medicine as an antipyretic or analgesic by Von Mering in 1893. Paracetamol has been used as an analgesic for home remedies and has been accepted for more than 30 years as a very effective treatment for relief of pain and fever in adults and children. Paracetamol is very useful in the after-treatment of acetylsalicylic acid in many countries as an alternative to aspirin and phenacetin (Lavent & Yucel, 2007). However, in recent studies, it has been shown that paracetamol is associated with hepatic toxicity and renal impairment even though paracetamol is non-destructive. At normal medicinal doses, very rapidly and completely metabolized paracetamol undergoes glucuronidation and sulfation of the slow metabolites and is excreted in the urine (Bosch et al., 2006). So it is necessary to measure the presence of paracetamol with precision so as not to harm the patient.

Many studies have been carried out to measure the presence of paracetamol using electrodes, such as research to determine paracetamol on various types of electrodes, namely by using graphite-based electrodes: carbon nanotubes (Moghaddam et al., 2010), polypyrrole modified glassy carbon electrodes (Muralidharan, Gopu, Vedhi, & Manisankar, 2009), MWNT film-coated electrodes (Wan et al, 2009), and graphene-modified glassy carbon electrodes (Kang et al., 2010). Meanwhile, the use of aminophenols in the manufacture of sensors has also been carried out for other compounds, namely electrodes modified with poly(o-aminophenol) thin film (Guerrieri, Ciriello, & Centonze, 2009) and copper modified gold electrodes on polymerized o-aminophenol films (Pan et al., 2005). For the use of aminophenols in the manufacture of MIP sensors from aminophenols, it has also been carried out for different compounds, namely the determination of dopamine based on the MIP of o-aminophenol (Li, Zhao, & Wei, 2009). While the use of MIP sensors to detect paracetamol has been published in the use of MIP polypyrrole modified graphite pencil electrodes (Lavent & Yucel, 2007). However, none of these studies have used MIP-modified graphite pencil electrodes from 3-aminophenol monomers as sensors to determine the presence of paracetamol. The use of Aminophenols monomers is an interesting electrochemical material because this material shows 2 groups that can be oxidized (NH_2 and OH). This material can exhibit

electrochemical behavior that mimics aniline and phenol. This upper binding group with polymeric molecules may be reactive sites for incorporation of metals or biomolecules, with applications in electrocatalysis and biosensors (Franco et al., 2008).

Detection of the presence of paracetamol in this study using molecularly imprinting technology. In this research, the 3-aminophenol monomer is used which will be the design of molecularly imprinted polymers. The process of forming poly (3-aminophenol) from 3-aminophenol monomer using electropolymerization technique. The molecule that will be used as a template is paracetamol. The calculation of the presence of paracetamol in this study used the differential pulse voltammetry technique. Therefore, this study aims to develop a modified carbon paste electrode with MIP poly (3-aminophenol).

EXPERIMENTAL SECTION

Materials and Tools

The materials used in this research are graphite powder (Fisher, American), liquid paraffin (Merck) which is used for the manufacture of carbon paste. 3-aminophenol solid (Merck, Germany), concentrated $HClO_4$ solution (Merck, Germany), solid NaH_2PO_4 (Merck, Germany), solid Na_2HPO_4 (Merck, Germany) for making phosphate buffer solutions pH 7, NaOH (Merck, Germany), HCl 37% (Merck, Germany) for adjusting the pH of the solution, ascorbic acid solids (Merck, Germany) and paracetamol solids (Merck, Germany).

The equipment used in this research is special equipment, namely eDAQ Potentiostats with ECem v2.1.0 software for paracetamol analysis using cyclic voltammetry and differential pulse voltammetry using three types of electrodes. The electrodes used were modified carbon paste electrodes (1.5 mm in diameter) which had been made as working electrodes, Ag | AgCl (NaCl 3M) electrodes as a reference electrode, and platinum electrodes as auxiliary electrodes. All solutions were entered into a 25 mL measuring cell.

ELECTRODE MANUFACTURING METHODS

Making Carbon Paste Electrodes

A copper wire with a plastic sheath of 2.5 mm in diameter is cut about 6 cm. The wire is then inserted into the electrode body made of glass and left a little space for the paste and then glued. The graphite powder and liquid paraffin in a ratio of 70:30 were then placed on a watch glass to be heated at 80°C until a paste was formed. The paste formed is compacted on the electrode body and the surface is smoothed by rubbing it on clean paper.

Making NIP and MIP Electrodes

The carbon paste electrode is modified with polyaminophenol (poly (3-aminophenol)). The varied electrode modifications were carried out on 2 electrodes, namely: (1). For the manufacture of the MIP working electrode, namely using the carbon paste electrode coated with the cyclic voltammetry methode in a solution of 0.02 M 3-aminophenol monomer and 0.02 M paracetamol with 0.1 M $HClO_4$ supporting electrolyte, 5 cyclics potential range −0.2V-1.2V with scan rate: 100 mV /sec. For the removal of paracetamol from the working electrode by conditioning it in 0.1 M KCl solution added with 0.1 M phosphate buffer solution (pH 7). Cyclic voltammetry was performed over a potential range of -0.6V – 1V as much as 20 cycles with a scan rate of 100 mV/sec. (2). For the next electrode modification is a carbon paste electrode coated with poly (3-aminophenol) in the absence of paracetamol. The modification of the carbon paste electrode was carried out by cyclic voltammetry in a 0.02 M 3-aminophenol monomer solution with 0.1 M $HClO_4$ support electrolyte in the absence of paracetamol. Cyclic voltammetry for coating with the 3-aminophenol monomer was carried out at a potential of −0.2V-1.2V as much as 5 cycles with a scan rate of 100 mV/sec.

Testing of carbon paste electrodes, polyaminophenol coated carbon paste electrodes and MIP electrodes in paracetamol solution.

Test of 1 mM paracetamol compounds in solution was carried out with modified working electrodes (NIP and MIP) and carbon paste electrodes with platinum electrodes as auxiliary electrodes and Ag | AgCl as a reference electrodes. To obtain a concentration of 1 mM paracetamol, the paracetamol solid was weighed as much as 0.1888 g and then dissolved in distilled water. The solution was transferred to a 25 mL volumetric flask and then diluted to the mark. From this treatment, a concentration of 0.05 M paracetamol was obtained, then 0.5 M paracetamol solution that had been made was taken and 12.5 mL of 0.1 M phosphate buffer (pH 7) solution was put into a 25 mL volumetric flask and diluted to the mark limit.

The voltammogram obtained from the varied working electrode clusters were measured the peak current and peak potential to determine the electropolymerization process that occurred from the 3-aminophenol monomer on the MIP electrode. Differential pulse voltammetry measurement data is carried out to determine the presence of paracetamol in the solution by looking at the response to changes in conditioned current at a scan rate of 10 mV/sec, 50 ms pulse interval, and 25 mV modulation amplitude.

RESULT AND DISCUSSION

Cyclic Voltammetry Study on Molecularly Imprinted Poly (3-amoniphenol) Electromerization.

In this study, 2 types of working electrodes were made, namely MIP and NIP with the cyclic voltammetry method at a potential of -0.2V-1.2V (versus Ag | AgCl) with a potential scan rate of 100

mV/sec for 5 cycles. (The use of this potential is so that the 3-aminophenol monomer is oxidized so that it can be deposited on the surface of the carbon paste electrode and this is also by the journals related to this study. The difference of these 2 electrodes can be seen from the solution used. Whereas in the manufacture of the MIP electrode, the solution consists of a 0.02 M 3-aminophenol monomer solution, 0.01M paracetamol, and 0.1 M $HClO_4$. While ~~for~~ on the manufacture of the NMIP electrode, the solution only consists of 0.02 M 3-aminophenol monomer solution and $HClO_4$ 0.1 M without adding paracetamol. The difference of voltammogram can also be seen from the number of peaks produced at the beginning of the cyclic. There is an oxidation peak of poly(3-aminophenol) at 2 voltammograms, but after the next cyclic in the voltammogram image (a) there is no peak while in the voltammogram image (b) there is a peak lower than the peak on the first cyclic which is most likely the peak of paracetamol trapped on the poly(3-aminophenol) matrix. The anodic peak of 3-aminophenol has been observed at the peak potential of 0.948 V, where the reduction process cannot be observed on a cyclic voltammogram so this is an irreversible reaction. The Cyclic voltammogram electropolymerization process without and with paracetamol (target molecules) can be seen easily in **Figure 1a** and **1b**. The peak oxidation potential of poly (3-aminophenol) shifted more to the cathodic potential, from 0.948 V to 0.780 V, which was due to the presence of paracetamol. This oxidation peak indicates that the target molecule has become part of the polymeric chain.

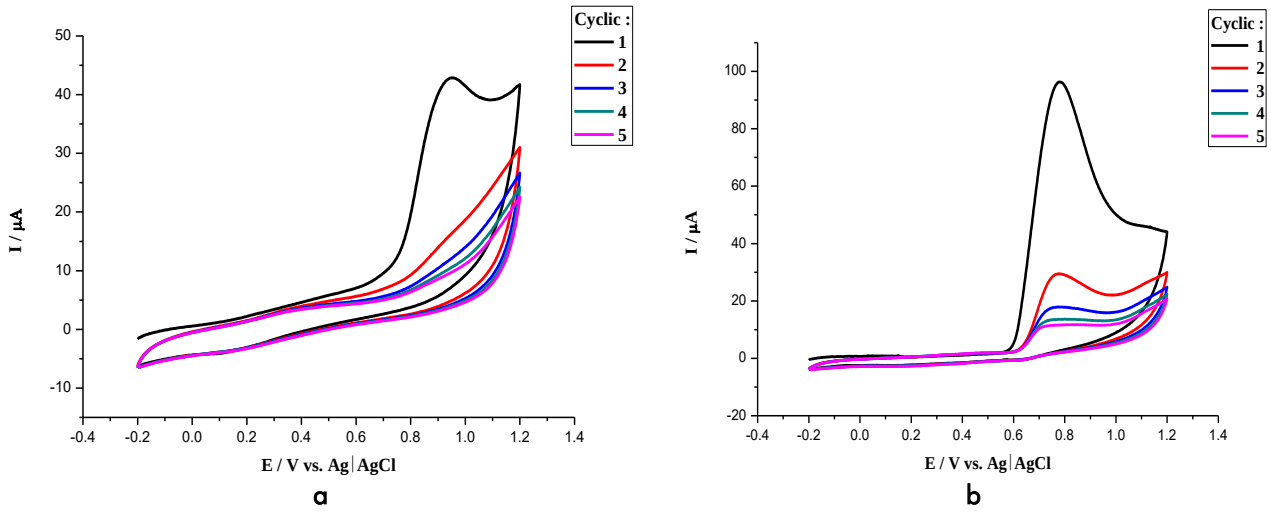


Figure 1. Cyclic voltammogram during the 3-aminophenol (0.02M) electropolymerization process. Cyclic voltammogram (**a**) without and (**b**) with paracetamol (0.01M) on the surface of the carbon paste electrode (potential scan rate: 100 mV/sec; supporting electrolyte $HClO_4$ 0.1 M; number of cyclics: 5)

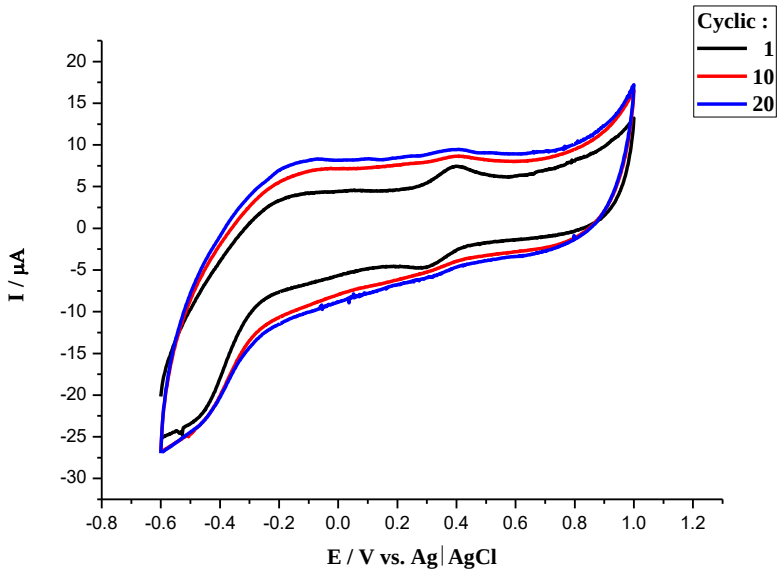


Figure 2. Cyclic voltammogram during the removal of peracetamol in the polymer at the MIP electrode in 0.1 M KCl solution added with 0.05 M phosphate buffer solution pH 7 (Number of cyclics: 20; scan rate: 100 mV/sec).

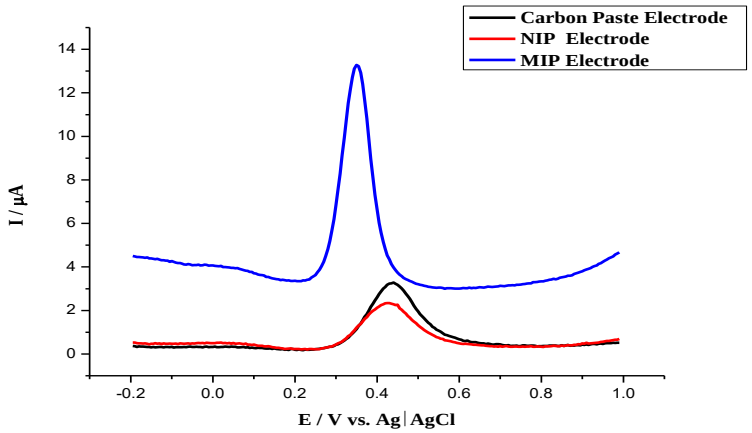


Figure 3. Differential pulse voltammogram at 1 mM paracetamol in phosphate buffer solution (pH 7).

After the electropolymerization process of 3-aminophenol in the manufacture of MIP electrodes, the paracetamol was trapped in the polymer matrix. Then the paracetamol trapped in the polymer matrix must be removed from the polymer matrix so that a mold of paracetamol is formed on the MIP electrode, so that the MIP electrode can be used to detect the presence of paracetamol. The removal of the trapped target molecules can be carried out by various types of possible methods namely extraction, oxidation-reduction of the template in the polymer, or the use of a strong solvent to interact with the polymer to release the template in the polymer. In this study, the removal of paracetamol was carried out by performing cyclic voltammetry in a supporting electrolyte solution of 0.1 M KCl mixture and 0.05 M phosphate buffer (pH 7). The obtained cyclic voltammogram during removal of peracetamol is shown in **Figure 2**. It was seen that the paracetamol peak decreased with the increasing number of cycles indicating the release of paracetamol from the electrode surface.

Testing the Presence of Paracetamol on 3 Electrodes by Electrochemistry

This test is intended to determine the ability of the MIP (Molecularly Imprinted Polymers), NIP (Non Molecularly Imprinted Polymers), and CPE (carbon paste electrode) electrodes made in detecting the presence of paracetamol. From the results of the research to detect 1 mM paracetamol in a phosphate buffer solution (pH 7) which can be seen in **Figure 3** and satisfactory results were obtained by obtaining the peak current on the differential pulse voltammogram for the presence of paracetamol. From the differential pulse voltammogram obtained, it is known that the MIP electrode shows better ability and sensitivity when compared to the NIP electrode and the carbon paste electrode, which can be seen from the current and potential obtained in the voltammogram. The peak of current paracetamol oxidation at the MIP electrode is 12.30 µA, while for the NIP electrode and the carbon paste electrode, it is 2.07 µA and 2.98 µA. The significant increase in peak current of the MIP electrode compared to the NIP electrode and the carbon paste electrode may be due to the specific ability of the MIP electrode to recognize the target molecule, namely paracetamol, where the target molecule can diffuse to the electrode surface through the pores of poly(3-aminophenol) and eventually undergo a redox reaction (Zhou et al., 2017). The oxidation potential of the potential is 0.355V while the NIP electrode and the carbon paste electrode are 0.425V and 0.440V. The oxidation potential of paracetamol on the MIP electrode has a more negative value compared to the other two electrodes, which indicates that paracetamol can be oxidized more easily on the surface of the MIP electrode (Mulyasuryani et al., 2019).

Effect of pH of Solutions in Electrochemical Paracetamol on MIP Electrodes

The effect of solution pH in the oxidation of paracetamol on the MIP electrode has been studied with a differential pulse voltammogram in a buffer solution with a pH range of 3-8, **Figure 4**. From the differential pulse voltammogram, it can be seen that the anodic peak potential shifts to a negative direction with the increasing pH of the solution. This indicates that the paracetamol oxidation reaction at the MIP electrode is influenced by pH in the paracetamol solution. The relationship between the peak current of oxidation and pH can be seen in **Figure 5b**. Meanwhile, the relationship between the oxidation peak potential and the pH value of the solution can be seen in **Figure 5a**, which shows the linear regression equation at Epa (V) = 0.725-0.052 pH, with the correlation constant R^2 = 0.995. The slope value obtained is -52 mV / pH unit, which is close to the theoretical value of -59 mV. This indicates that the number of protons and electrons involved in the oxidation mechanism is the same. The paracetamol oxidation process yields two electrons and the number of protons involved is predicted to be two (Rosi et al., 2015; Zhou et al., 2017 & Farida et al., 2019). The equation for the reaction that occurs can be written as follows:

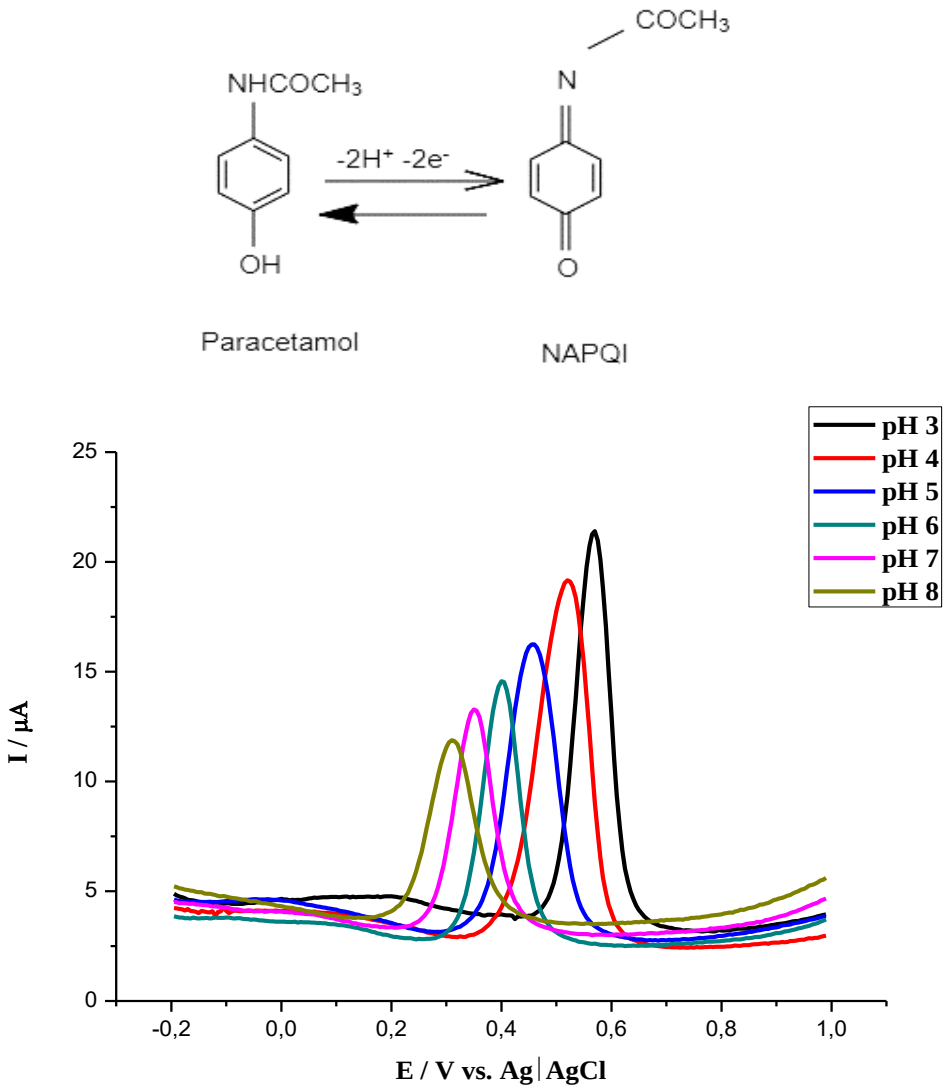


Figure 4. Differential pulse voltammogram for the effect of pH on the response of the MIP electrode at 1 mM of paracetamol in phosphate buffer solution.

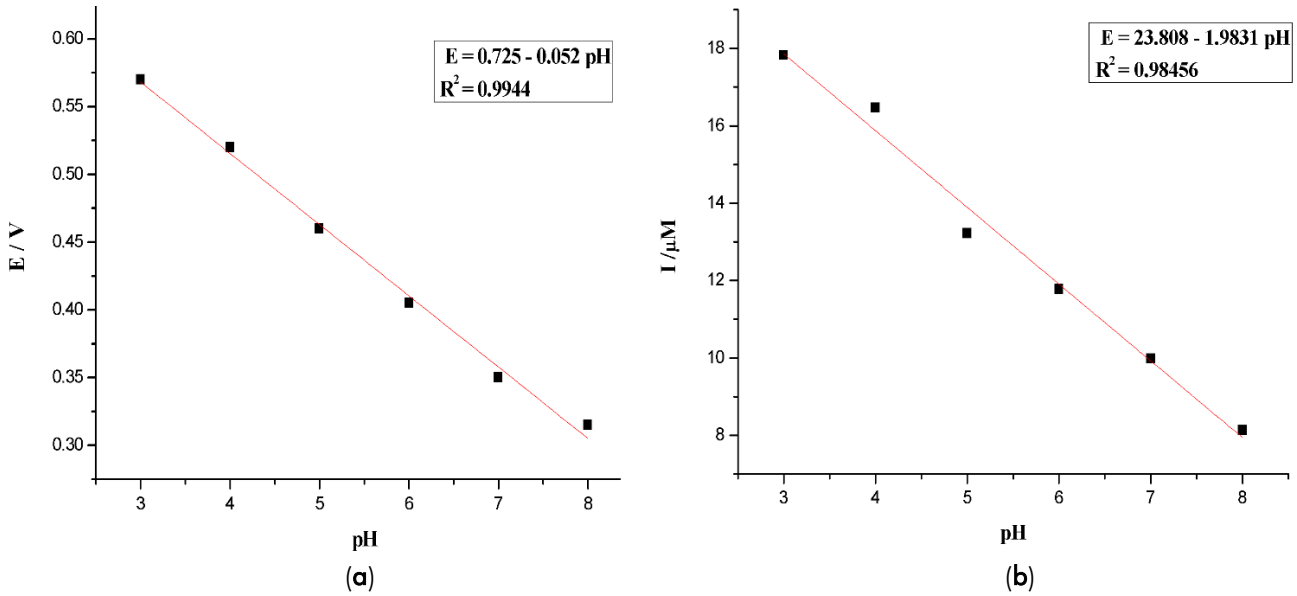
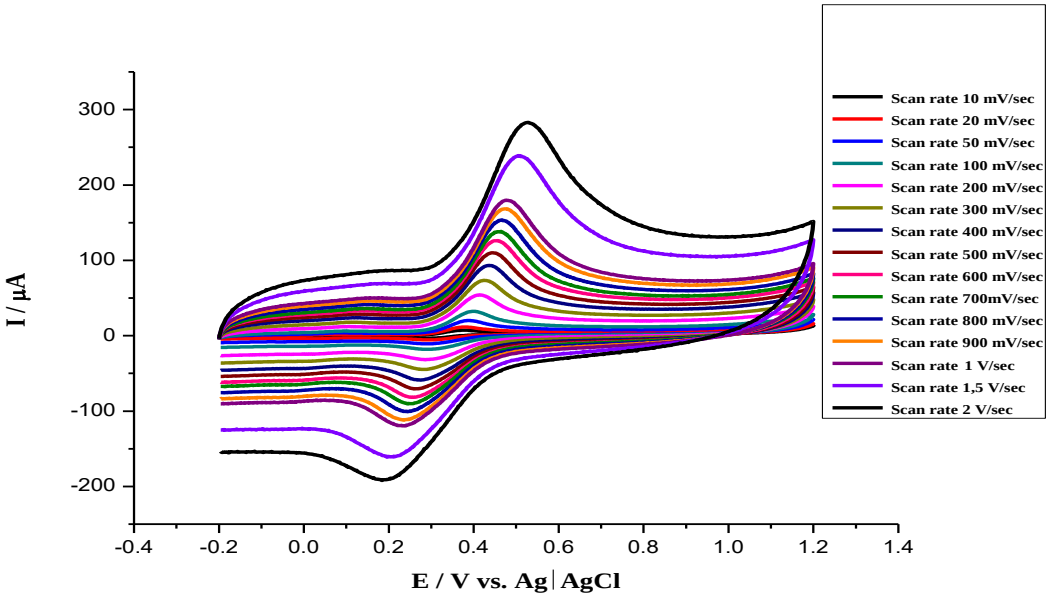


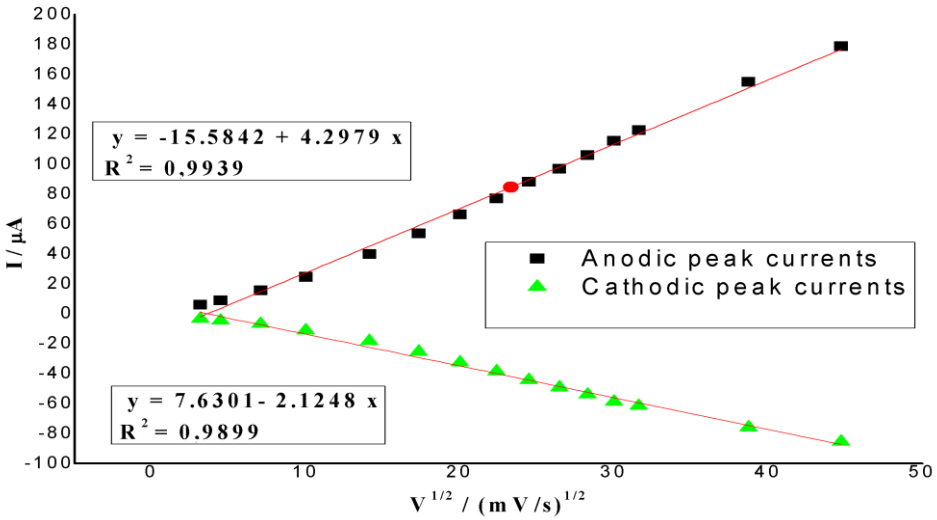
Figure 5. Effect of solution pH on (**a**) potential and (**b**) current on the MIP electrode response to 1 mM paracetamol in phosphate buffer solution.

Effect of Potential Scan Rate

The effect of the potential scan rate on the peak currents of oxidation and reduction in 1 mM paracetamol on the surface of the MIP electrode in phosphate buffer solution (pH 7) was studied in this research. **Figure 6** shows a cyclic voltammogram of paracetamol over a varied range of potential scan rates. **Figure 6a**. produces anodic and cathodic peak currents which confirm that a reversible process has occurred from the paracetamol solution on the surface of the MIP electrode. The redox peak current at the MIP electrode in paracetamol solution increases linearly with the potential scan rate at the limit of 10 to 2000 mV/sec which is related to the observation of the presence or absence of the redox process of paracetamol, and linked to the diffusion process of paracetamol in solution or adsorption on the MIP electrode. Potential values (*Epa* and *Epc*) in this study is 0.20 V and 0.45 V, where *Epa* and *Epc* values experience a potential shift as the scan rate increases used in the paracetamol analysis process. Large and small potential values are influenced by electron transfer kinetics, if the electron transfer kinetics is slow, the magnitude of the peak potential separation will be greater andm increase in accordance with the increase in scan rate (Farida et al., 2019). The current peak of paracetamol redox shows a linear relationship with the increase in scan rate after rooting ($\upsilon^{1/2}$) which can be seen in **Figure 6b**. it indicates that there has been a diffusion control process for paracetamol electrooxidation.

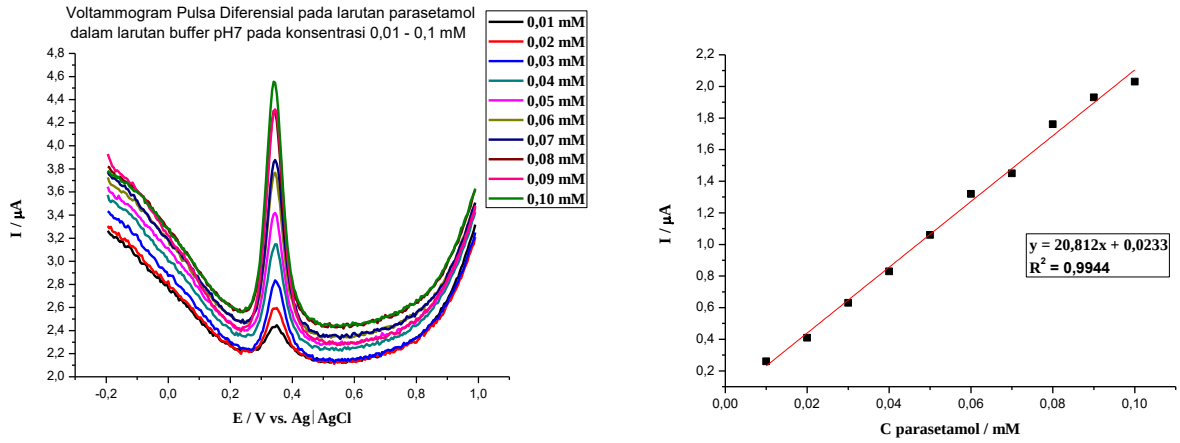


(a)



(b)

Figure 6. (**a**) Cyclic voltammogram on the MIP electrode with 1 mM paracetamol in a phosphate buffer solution (pH 7) at different potential scan rates from 10 to 2000 mV/sec. (**b**) Plot of the anodic peak currents and the cathodic peak currents versus the roots of the potential scan rates of 10-2000 mV/sec.



(a) (b)

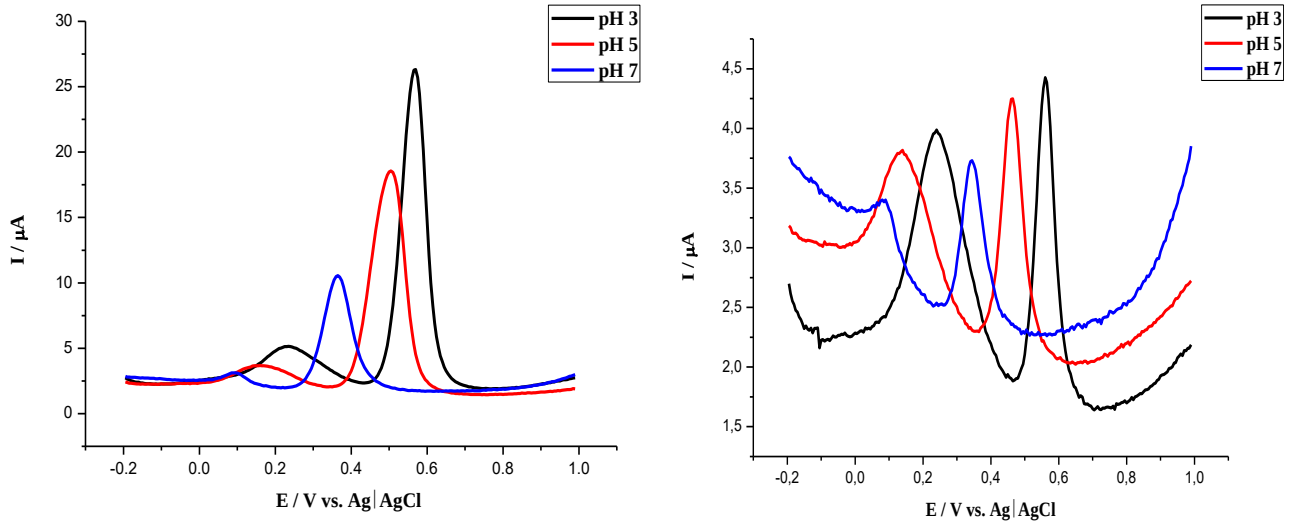
Figure 7. (a) Differential voltammogram on paracetamol solution in phosphate buffer solution (pH 7) for concentration range 0.01-0.1 mM. (b) Linear relationship between paracetamol concentration and peak current.

Linear Range and Detection Limit

The peak current was found to be regression linearly dependent on Paracetamol concentration in the range 0.01–0.1 mM (**Figure 7b**). The regression equation was I_{pa} = 20.812c + 0.0233, R^2 = 0.9944. According to S/N = 3, the detection limit was calculated to be 4,63 μM.

Effects of Interferense Compounds

The selectivity and sensitivity of the MIP electrode in this study for the determination of the mixture of paracetamol and ascorbic acid were evaluated. **Figure 8** shows the reported differential pulse voltammogram for (a) a mixture of ascorbic acid (1 mM) and paracetamol (1 mM) while for (b) a mixture of ascorbic acid (1mM) and paracetamol (0.1 mM). It can be seen that the MIP electrode can properly separate ascorbic acid and paracetamol at a solution pH of 3, 5, and 7. The peak potential for oxidation of ascorbic acid (1 mM) and paracetamol (1mM) at the pH of solution 3 were 0.235 V and 0.570 V. Mean while for the pH of solution 5 were 0.160 V and 0.505 V, respectively and for solution pH 7, respectively are 0.095 V and 0.365 V. For a mixture of ascorbic acid (1 mM) and paracetamol (0.1 mM) at pH 3, the oxidation potential peaks are 0.24 V and 0.56 V, respectively. Respectively, were 0.140 V and 0.465 V. Finally, the pH of solution 7 was 0.090 V and 0.345 V, respectively. From the research, it is known that the presence of ascorbic acid is not too disturbing in the measurement in paracetamol solution even though the concentration ratio between paracetamol and ascorbic acid is 1:10.



(a) (b)

Figure 8. Differential pulse voltammogram for the presence of 1 mM ascorbic acid in (a) 1 mM paracetamol and (b) 0.1 mM paracetamol on the MIP electrodes in a phosphate buffer solution (pH 7).

CONCLUSIONS

In this study, the MIP electrodes that have been made have successfully detected the presence of paracetamol. The MIP electrodes that have been made provide better potential and peak current responses compared to the NIP electrodes and carbon paste electrodes. The pH of the solution in the paracetamol sample affects the potential and peak currents of paracetamol, the greater the pH, the more potential shifts to a more negative direction, and the lower the peak current. The peak potential for oxidation of ascorbic acid (1 mM) and paracetamol (1mM) at the pH of solution 3 were 0.235 V and 0.570 V. Mean while for the pH of solution 5 were 0.160 V and 0.505 V, respectively and for solution pH 7, respectively are 0.095 V and 0.365 V. For a mixture of ascorbic acid (1 mM) and paracetamol (0.1 mM) at pH 3, the oxidation potential peaks are 0.24 V and 0.56 V, respectively. Respectively, were 0.140 V and 0.465 V. Finally, the pH of solution 7 was 0.090 V and 0.345 V, respectively. Thus, MIP electrodes can provide significant peak differences in the mixed solution consisting of paracetamol and ascorbic acid. To better understand the effect of electropolymerization in the manufacture of MIP for paracetamol analysis, it is necessary to study other aminophenol monomers, namely 2-aminophenol and 4-aminophenol. Sensors obtained from carbon paste electrodes modified with MIP poly(3-aminophenol) have better linear range and detection limit. Rregression linearly dependent on Paracetamol concentration in the range 0.01–0.1 mM with regression equation was $I_{pa} = 20.812c + 0.0233$, $R^2 = 0.9944$ and the detection limit was calculated to be 4,63 μM . Thus, this electrode is highly recommended for its practical application, the MIP electrode is expected to be used in the future for determination and quantification in the pharmaceutical industry as well as for medical purposes.

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