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Full Paper

A New Sensor for Determination of Paracetamol, Phenylephrine Hydrochloride and Chlorpheniramine Maleate in Pharmaceutical Samples Using Nickel Phosphate Nanoparticles Modified Carbon Past Electrode

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Abstract- A new chemically modified electrode was constructed based on nickel phosphate nanoparticles modified carbon paste electrode that immersed in $NiCl_2$ solution (Ni-NP/CPE). The modified electrode was employed as a sensor for electrocatalytic oxidation of paracetamol (PAR), phenylephrine hydrochloride (PHE), chlorpheniramine maleate (CLP) in aqueous solutions using differential pulse voltammetry (DPV) method in Tris-HCl buffer solution with pH 7.0. The Ni(II) ion adsorbed in nickel phosphate nanoparticles can act as catalyst to oxidize above drugs. The prepared electrode showed voltammetric responses with high sensitivity and selectivity for PAR, PHE and CLP in optimal conditions, which makes it very suitable for determination of these compounds. A linear calibration graph was obtained with concentration ranges of 0.75–7.0, 0.02–10.0 and 0.05–10.0 mM for PAR, PHE and CLP, respectively. The detection limits of PAR, PHE and CLP were obtained 0.24, 0.0064 and 0.016 mM, respectively. The proposed method was successfully applied for determination of PAR, PHE and CLP in pharmaceutical samples.

Keywords- Modified Carbon Paste Electrode, Nickel Phosphate Nanoparticles, Paracetamol, Phenylephrine Hydrochloride, Chlorpheniramine Maleate, Differential Pulse Voltammetry

1. INTRODUCTION

A mixture of paracetamol (PAR), phenylephrine hydrochloride (PHE) and chlorpheniramine maleate (CLP) is widely used in diseases accompanied by cough, pain and fever such as the common cold and other viral infections as an analgesic, antipyretic, decongestant, antihistamine and antitussive [1]. PAR (*N*-acetyl-*p*-aminophenol) is a commonly used analgesic and antipyretic drug [2]. Its structure is shown in Fig. 1a that oxidizes to *N*-acetyl-*p*-quinoneimine in neutral media [3]. Several analytical techniques such as spectrophotometry [4], spectrofluorometry [5], chromatographic [6–9], colorimetry [10], amperometry [11], voltammetry [12, 13] and chemometric [14] methods are proposed for the determination of PAR in pharmaceutical formulations and biological samples. Voltammetric determination of PAR at chemically modified electrodes [15,16] and screen-printed electrodes [17] have also reported. Ozcan et al. [18] was reported a differential pulse voltammetry to determine the PAR using pencil graphite electrode prepared by imprinting electropolymerization. Babaei et al. [19] reported a sensor for the determination of PAR and mefenamic acid using Cu(II) doped into NaY zeolite modified carbon paste electrode. Goyal et al. [20] was used a nanogold modified indium tin oxide electrode for the determination of PAR using differential pulse voltammetry.

PHE ((*R*)-3-hydroxy-*α*-[(methylamino) methyl] benzenemethanol hydrochloride) is a sympathomimetic drug used for nasal congestion, sinusitis and rhinitis, its structure is shown in Fig. 1b [21]. The most recent methods for determination of PHE included chromatographic [22–25], electrochemical [26,27], spectrophotometric [28–30] and chemometric [14,31] techniques. CLP is chemically 2-pyridinepropanamine, *γ* (4-chlorophenyl)-*N*,*N*-dimethyl, (*Z*)-2-butenedioate that its structure is shown in Fig. 1c. It is an antihistamine drug and is widely used as an ingredient in antitussive formulations [1]. These compounds are the pharmacologically active constituents found in most cough-cold syrups [32]. Several methods have been reported for determination of CLP such as chromatographic [21, 33–35], spectrophotometric [36,37] and chemometric [14,31] techniques.

Due to the simple preparation and easy surface refreshing of the carbon past electrodes (CPEs) have been used extensively as working electrodes for a variety of electrochemical applications. It has been shown that carbon, in comparison to other commonly used electrode materials, has more compatibility with biological samples [38]. Among of electrodes, CPEs have been widely used as suitable matrices for preparation of the modified electrodes because of the simplicity of their construction, easy renewability of the surface and compatibility with various types of modifiers [39]. Design of modified electrodes for electrocatalysis has been extensively developed because it provides an excellent way to facilitate (accelerate) charge transfer processes [40]. This contributes to decrease the overpotentials which often required to perform electrochemical transformations, as well as to enhance the intensity of the corresponding voltammetric responses. Nanocrystal materials can shorten the diffusion paths

for reactant and product molecules and ultimately, the undesired diffusion limitations of heterogeneous reactions can be reduced or eliminated [41]. These materials can use for modification of CPE.

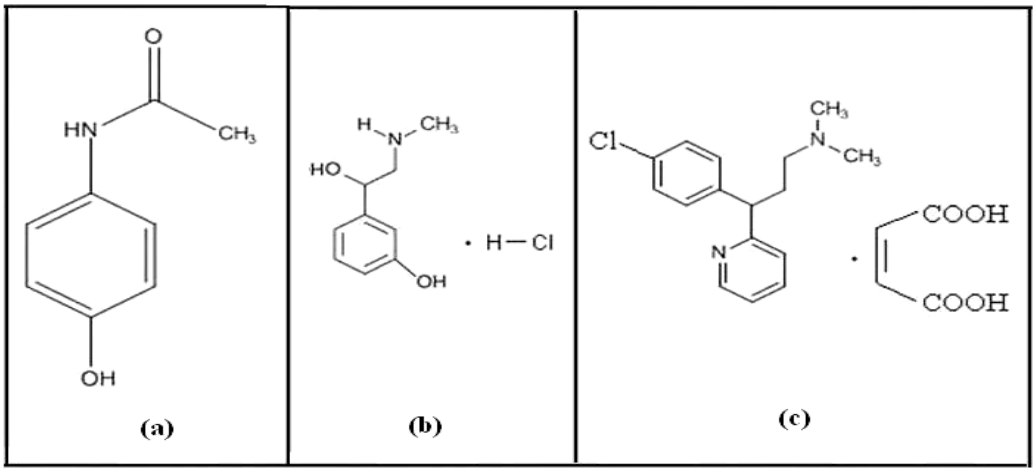

Fig. 1. Molecular structures of the (a) paracetamol, (b) phenylephrine hydrochloride and (c) chlorpheniramine maleate

Two of us reported a new method for the simultaneous determination of PAR, PHE and CLP using multivariate calibration-1 techniques [14]. All of three compounds are electroactive that can be oxidized electrochemically. Since voltammetric techniques are more selective, less costly and less time-consuming, they are widely used for the determination of drugs in pharmaceutical preparations. In respect of literature survey, there has been no report use of an electrochemical sensor with nickel phosphate nanoparticles modified carbon paste electrode for determination of PAR, PHE and CLP. In this study for the first time, we present the modification of carbon paste electrode with nickel phosphate nanoparticles and it used for determination of PAR, PHE and CLP using differential pulse voltammetry (DPV) method.

2. EXPERIMENTAL

2.1. Reagents and solutions

Paracetamol (PAR), Phenylephrine hydrochloride (PHE) and Chlorpheniramine maleate (CLP) were purchased from Sigma-Alderich and used without further purification. All stock solutions were freshly prepared by dissolving 25 mg of the three compounds in 5 ml of double distilled water. The concentrations of PAR, PHE and CLP were adjusted 3.307×10^{-2}, 4.664×10^{-2} and 1.279×10^{-2} mol L^{-1}, respectively. All PAR, PHE and CLP solutions were prepared by diluting stock solutions with double distilled water. Doubly distilled water was

used thoroughly. Nickel phosphate nanoparticles were synthesized in our laboratory and reported elsewhere [41].

2.2. Instrumentation

The electrochemical experiments were performed at room temperature using Palmsence electrochemical analyzer system with a voltammetry cell in a three electrodes configuration. A platinum wire and Ag|AgCl|KCl (3 M) were used as auxiliary and reference electrodes, respectively. The modified carbon paste electrodes with nickel phosphate nanoparticles (NP/CPE) were used as working electrode. The Differential pulse voltammograms was obtained for different drug solution in Tris-HCl buffer (0.01 M, pH 7 at 25 ºC) with pulse amplitude of 25 mV, scan rate of 20 mVs^{-1}, pulse interval of 0.3 S and step potential of 0.005 V. A Metrohm 710 pH meter was used for pH adjustments.

2.3. Preparation of nickel phosphate nanoparticles-modified carbon paste electrode

The modified carbon paste electrodes (NP/CPE) was prepared by mixing 10 mg of nickel phosphate nanoparticles (NP) with 60 mg of graphite, and then paraffin oil was added drop-wise until a uniform paste was obtained. This paste was deposited into a cavity, with a copper wire as electrical contact. This proportion was used because a better response was obtained in a preliminary test, however, a more detailed study about the paste composition should be considered. The second modified electrodes namely (Ni-NP/CPE) was prepared by immersing NP/CPE electrodes in the solution of 0.1 M $NiCl_2$ with stirring for five minutes at 200 rpm, and then washed completely with distilled water.

2.4. General procedure

Solutions (10 mL) containing appropriate amounts of each drug in 0.01 M Tris-HCl at pH 7 was transferred into the voltammetric cell. The differential pulse voltammograms were recorded by applying positive-going potentials from –0.3 to 0.3 V. The heights of anodic peaks currents were proportional to concentrations of drugs in solution. The calibration curves were obtained by plotting anodic peak currents of each drug versus the corresponding concentrations. All experiments were carried out under open circuit conditions. After each measurement, the Ni-NP/CPE was regenerated by pushing an excess of paste out of the tube, removing the excess, mechanically polishing the electrode surface and immersing of NP/CPE electrodes in the solution of 0.1 M $NiCl_2$ for five minutes, and then washed completely with distilled water.

3. RESULTS AND DISCUSSION

3.1. Voltammetric characteristics of PAR, PHE and CLP

Modified electrodes with transition metal have the ability to catalyze the oxidation of solute species. The major effect of the modifier is to decrease the potential that requires for the catalyzed redox systems, which is generally accompanied with a considerable increase in sensitivity. Transition metal ions show electrocatalytic activity and have shown promise for the electrocatalytic determination of many organic and biologically important compounds [42,43]. Here, we used the nickel phosphate nanoparticles as a modifier for modification of carbon paste electrode as a differential pulse voltammetric sensor to study the electrocatalytic oxidation of Paracetamol (PAR), Phenylephrine hydrochloride (PHE) and Chlorpheniramine maleate (CLP).

The differential pulse voltammograms of PAR, PHE and CLP at bare carbon paste electrode (CPE), nickel phosphate nanoparticles modified carbon paste electrode (NP/CPE) and nickel phosphate nanoparticles modified carbon paste electrode that immersed in 0.1 M $NiCl_2$ (Ni-NP/CPE) are illustrated in Fig. 2. Curves (A), (B) and (C) in this Fig shows the voltammograms of 7.0 mM of each drug at (a) CPE, (b) NP/CPE and (c) Ni-NP/CPE for PAR, PHE and CLP, respectively. As can be seen in Fig. 2 a very small oxidation peak is observed for PAR, PHE and CLP in bare CPE. Slight enhancements of the oxidation peak currents for all three compounds were observed at the NP/CPE. The oxidation peaks were observed at the peak potential of –0.061, 0.004 and –0.106 V for PAR, PHE and CLP, respectively. The excellent improvement in oxidation peak currents for all drugs was observed at Ni-NP/CPE electrode. The oxidation peaks potentials shifted to the positive values at Ni-NP/CPE electrode and observed at –0.004, 0.009 and –0.006 V for PAR, PHE and CLP, respectively.

Refer to the previous report [19,44], it can be supposed that there is a tendency for complex formation between Ni(II) and PAR, PHE and CLP. The enhancement of current peak and a shift in the oxidation potential in the anodic direction are a clear evidence of the catalytic effect of the Ni-NP/CPE electrode towards the oxidation of all compounds. It is possible that more of these drugs could be accumulated on the surface of electrode due to their interaction with adsorbed Ni(II) ion on the surface of modified electrode. Therefore, the use of Ni-NP/CPE leads to enhancement of the current sensitivity and selectivity for the determinations of these drugs than use of NP/CPE or bare CPE.

Voltammograms (c) of each drug at Ni-NP/CPE electrode in Fig. 2 showed that the most peak current is attributed to the oxidation of PHE and anodic peak current for CLP is bigger than that for PAR. PAR is an amide meanwhile; PHE and CLP are secondary and tertiary amines, respectively. The alkalic strength of amides are lower than that for amines and affinity of complex formation between adsorbed Ni(II) ion on the surface of Ni-NP/CPE electrode (Lewis acid) and PAR (Lewis base) is weaker than that for PHE and CLP.

Therefore, less of PAR molecule accumulated on the surface of Ni-NP/CPE electrode and obtained anodic current for PAR is lower than two other compounds. The alkalic strength of CLP (tertiary amine) is smaller than that for PHE (secondary amine) and the other hand, two CH_3 groups on the aliphatic nitrogen in CLP is created more steric hinder than one CH_3 group on the aliphatic nitrogen in PHE (see Fig. 1). As a result, tendency of complex formation between adsorbed Ni(II) ion on the surface of Ni-NP/CPE electrode and CLP is weaker than that for PHE and obtained anodic peak current for PHE is bigger than that for CLP at the same concentration (i.e. I_{Pa} (PHE) > I_{Pa} (CLP) > I_{Pa} (PAR)).

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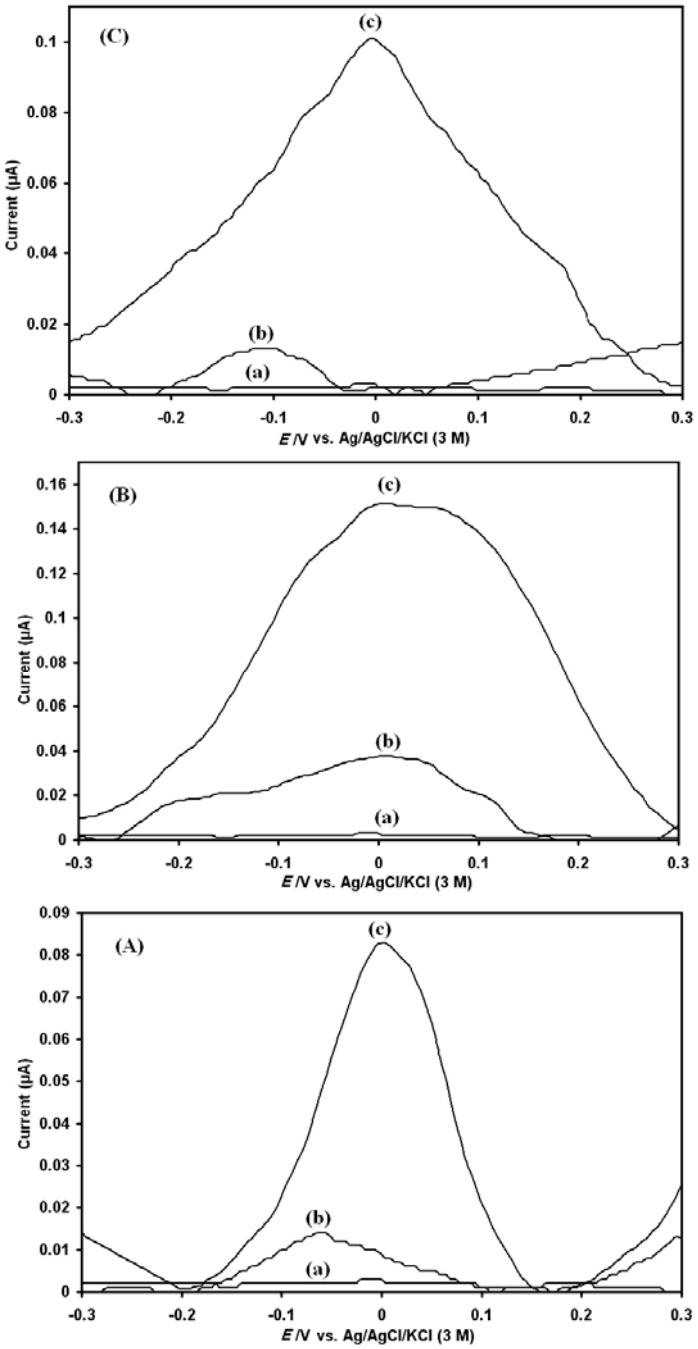

Fig. 2. Differential pulse voltammograms with concentration of 7.0 mM for (A) paracetamol, (B) phenylephrine hydrochloride and (C) chlorpheniramine maleate in Tris-HCl buffer (pH of 7.0) at (a) bare carbon paste electrode (CPE), (b) nickel phosphate nanoparticles modified carbon paste electrode (NP/CPE) and (c) nickel phosphate nanoparticles modified carbon paste electrodes that immersed in 0.1 M of $NiCl_2$ solution (Ni-NP/CPE)

3.2. Effect of operational parameter

Any variation of the accumulation potential has no significant effect on the proposed electrochemical sensor response, so all measurements were carried out at open circuit condition. In order to study the influence of accumulation time on electrochemical behavior of drugs, electrode was immersed in 0.1 M of $NiCl_2$ solution with different times. The applied time ranged between 10 and 500 s. The best time for accumulation was obtained 300 s (5 min). The different mass ratio between nickel phosphate nanoparticles and graphite powder was studied. The best answer (i.e. largest current) was for the 6:1 mass ratio of graphite powder and nanoparticles. Also, the effect of pH was studied, that the best response found in the pH of 7.0. On the basis of these results, the accumulation time of 5 min, the graphite powder-nickel phosphate nanoparticles mass ratio of 6:1 and 0.01 M Tris-HCl at pH of 7.0 were used for the following experiments.

3.3. Linear range, detection limit and reproducibility of the method

To verify the linear relationship between anodic peak currents and PAR, PHE and CLP concentrations, several calibration curves were constructed under optimum conditions in 0.01 M of Tris-HCl buffer (pH=7.0) solutions. Fig. 3(a-c) show calibration plots for PAR, PHE and CLP obtained at Ni-NP/CPE in various concentrations, respectively. A linear dynamic range from 0.75 to 7.0 mM, with a regression equation of I_p (μA) = 0.0049 C (mM) + 0.049 (R^2=0.9951), and a detection limit of 0.24 mM (*S*/*N*= 3) was obtained (see Fig.3a).

The calibration curve for the DPV oxidation peak current vs. concentration of PHE (see Fig.3b) shows in excellent linearity over a concentration range of 0.02 mM to 10.0 mM with a correlation coefficient of 0.9987 and can be expressed by the equation: I_p (μA) = 0.0132 C (mM) + 0.0587. The detection limit of PHE is 0.0064 mM.

A similar linear relationship was observed over the range of 0.05 to 10 mM for CLP. Here, a regression equation of I_p (μA)=0.0076 C (mM) + 0.0483 (R^2=0.9908) was obtained with a detection limit of 0.016 mM (see Fig. 3c). The linear behavior of the calibration curve further indicates that the process is basically diffusion controlled within the studied concentration range [20].

3.4. Effect of interferences

The effect of various components in the determination of PAR, PHE and CLP were studied by applying the method of mixed solutions. The influences of common interfering species were investigated for solutions of 7.0 mM of each drug under optimal conditions. When the developed procedure was exploded for the determination of 7.0 mM of each drug with optimum conditions, no interference was encountered for additions of 700 mM of each of following drug such as baclofen, metronidazole, nicotinamide, phenobarbital, tramadol and dectrometorphan hydrobromide. The tolerance limit was defined as the concentrations which give an error of ≤10% in the determination of PAR, PHE and CLP compounds. However, the

presence of 0.7 mM of aspirin or 1.4 mM of pyridoxine caused increase in the oxidation peak.

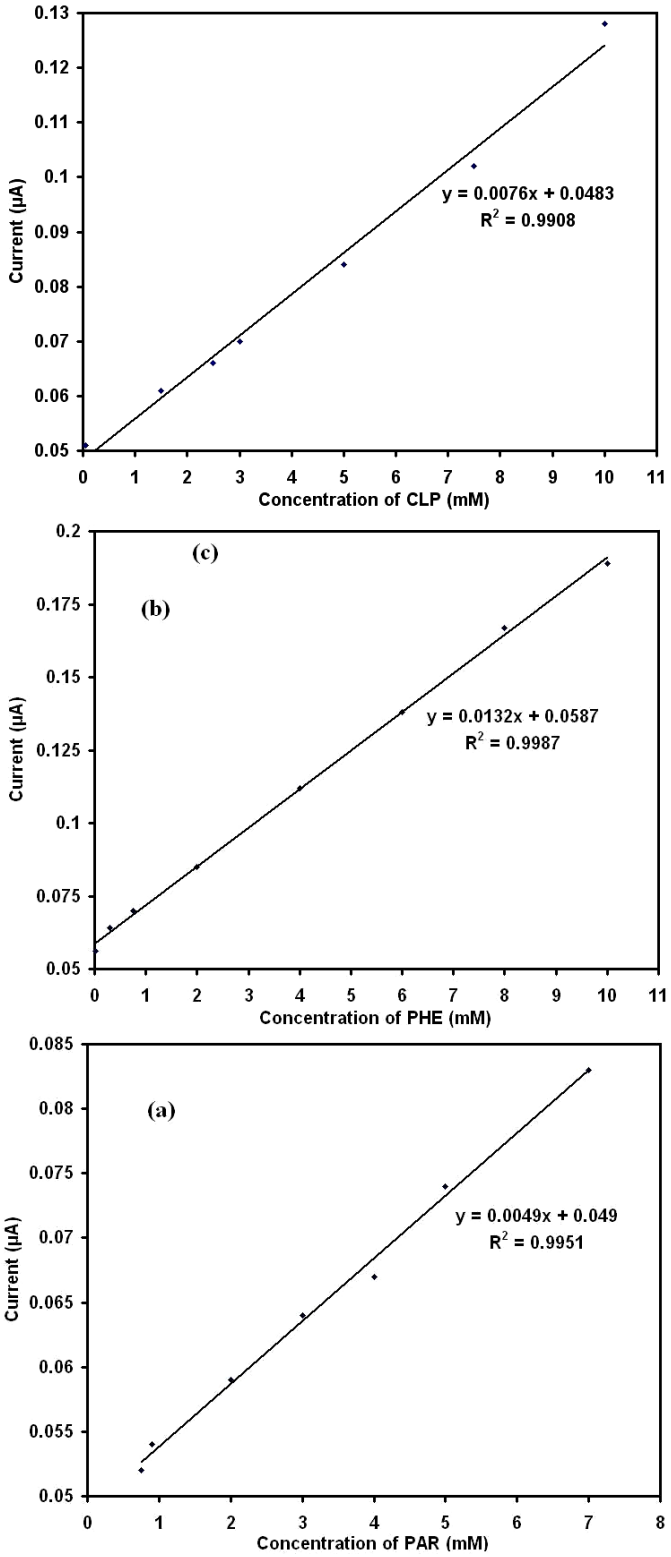

Fig. 3. Calibration plots observed at Ni-NP/CPE in buffer Tris-HCl (pH of 7.0) for (a) paracetamol, (b) phenylephrine hydrochloride and (c) chlorpheniramine maleate

3.5. Analysis of commercial samples

The applicability of the Ni-NP/CPE to the determination of PAR in pharmaceutical tablet and PHE and CLP in pharmaceutical ampoules was examined at optimum conditions by nickel phosphate nanoparticles modified carbon paste electrode. The PAR tablet was grounded to powder and then dissolved in double distilled water. Solution obtained by dissolution of PAR tablet and PHE and CLP ampoules were subsequently diluted so that concentration lies in the working ranges. The concentrations were obtained by applying calibration plot. Experimentally determined and reported amount of PAR, PHE and CLP are listed in Table 1. The results are in good agreement with the manufacturers' stated contents of PAR, PHE and CLP. The recoveries for the proposed method were acceptable, showing that the method could be efficiently used for the determination of these compounds in biological systems and pharmaceutical preparations.

Table 1. Determination of PAR, PHE and CLP in pharmaceutical preparations with Ni-NP/CPE

Analyte	Reported (mM)	Found (mM)	RSD (%)	Recovery (%)
PAR	1.10	1.15	1.84	104.5
	2.50	2.39	0.88	95.6
	4.30	4.46	1.01	103.7
PHE	2.45	2.49	1.28	101.6
	4.90	4.83	0.44	98.6
	6.12	6.05	0.46	98.8
CLP	2.75	2.63	2.33	95.6
	7.00	7.19	0.52	102.7
	8.50	8.62	0.25	101.4

4. CONCLUSION

Zeolite was found to have good adsorption characteristics and catalytic capabilities. This work demonstrated that Ni(II) loaded in nickel phosphate nanoparticles can catalytically oxidize Paracetamol (PAR), Phenylephrine hydrochloride (PHE) and Chlorpheniramine maleate (CLP). Determination of PAR, PHE and CLP was performed by Differential pulse voltammetry (DPV) at nickel phosphate nanoparticles modified carbon paste electrode. Results specified that tendency of complex formation between adsorbed Ni(II) ion on the surface of Ni-NP/CPE electrode and PHE is more favorable than that for CLP and PAR and obtained anodic peak current for PHE is the biggest that others at the same concentration (i.e. I_{Pa} (PHE) > I_{Pa} (CLP) > I_{Pa} (PAR)). This method was developed for the determination of

PAR, PHE and CLP in pharmaceutical samples. Results obtained from this study specified that the high recovery values for commercial samples indicate good accuracy of the method for all three compounds.

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