Journal of Applied Sciences

ISSN 1812-5654

science alert

ANSInet
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Journal of Applied Sciences 6 (7): 1625-1627, 2006
ISSN 1812-5654

Electrochemical Behavior and the Determination of Omeprazole Using Glassy Carbon Electrode

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Abstract: The electrochemical behavior of omeprazole on a glassy carbon electrode was investigated by cyclic voltammetry and differential pulse voltammetry. It was found that omeprazole would give a sensitive oxidation peak at +0.74 V in the HAc-NaAc buffer solution (pH 5.10) under the Differential Pulse Voltammetric (DPV) mode. The peak current was linear with the concentration of omeprazole in the range of 1.0~20 mg L^{-1}. Based on which, a DPV method for determination of omeprazole with the detection limit of 0.19 mg L^{-1} has been developed. The proposed method has been used for determination of omeprazole concentration in omeprazole enteric-coated tablets, the recovery was found to be in the range of 99.3~102%. The mechanism for this electrochemical reaction at the glassy carbon electrode was also discussed in this study.

Key words: Omeprazole, electrochemistry, glassy carbon electrode

INTRODUCTION

Omeprazole is used for the treatment of infection caused by *Helicobacter pylori*, it acts to regulate acid production in the stomach and is used to treat various acid-related gastrointestinal disorders. *Helicobacter pylori* urease belongs to a family of highly conserved urea-hydrolyzing enzymes. A common feature of these enzymes is the presence of two Lewis acid nickel ions and a reactive cysteine residue in the active site. The H^+/K^+-ATPase inhibitor omeprazole is a pro-drug of a sulfenamide which covalently modifies cysteine residues on the luminal side of the H^+/K^+-ATPase of gastric parietal cells. The inhibition was potentiated by a lower pH (favoring the formation of the sulfenamide) but abolished in the presence of beta-ercaptoethanol (Kuhler *et al*., 1995). Omeprazole is a benzimidazole compound, 5-methoxy-2-[[(4-methoxy -3, 5-dimethyl-2-pyridinyl)methyl]sulfinyl]-1H-benzimidazole (Fig. 1), that acts as proton pump inhibitor. Its empirical formula is $C_{17}H_{19}N_3O_3S$, with a molecular weight of 345.42.

Sporadic publications on the identification of Omeprazole by liquid chromatography (Schubert *et al*., 2003) HPLC (Sluggett *et al*., 2001), Spectrophotometric method (Chilukuri *et al*., 1997; Salama *et al*., 2003), capillary electrophoresis (Nevado *et al*., 2005) have appeared in the literature. In this research reported here the utility of electrochemical analysis method using glass carbon electrode as working electrode for the determination of omeprazole for the first time. In HAc-NaAc buffer solution (pH 5.10), a sensitive differential pulse voltammetric peak of Omeprazole at glass carbon electrode at about +0.74 V (vs Ag/AgCl) is found. The electrochemical behavior and reaction mechanism of this system have been studied by cyclic voltammetry, linear sweep voltammetry and differential pulse voltammetry. There is a good linear relationship between the peak current and the concentration of omeprazole in the range of 1.0~20 mg L^{-1}. The detection limit of the method is 0.19 mg L^{-1}. The electrochemical analysis method described here enables simple and rapid determination of omeprazole in real samples. The concentration of omeprazole in omeprazole enteric coated tablets has been determined with good results by this method.

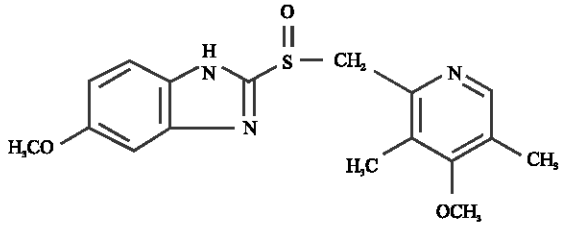

Fig. 1: Structure of omeprazole

MATERIALS AND METHODS

Apparatus and reagents: All measurements were carried out with a Model CHI832 multifunction voltammetric analyzer system (Shanghai Chenhua Electroanalysis Instruments Corporation, China). A glass carbon electrode with area 0.785 mm^2 was used as working electrode. An Ag/AgCl was used as a reference electrode together with a platinum wire as the counter-electrode. The pH measurements were carried out with a 25 pHS-2C model acidity meter (Leici Instrumental Factory, Shanghai, China), using a combination electrode. The electrolytic

cell was a 50 mL beaker. A SRD-1 Model magnetic stirrer and a stirring bar (2.5 cm) in length provided the convective transport during the pre-concentration. All experiments were performed at room temperature and dissolved oxygen was removed form the solutions by bubbling oxygen-free nitrogen through the cell for 10 min.

Omeprazole was obtained from Sigma and was used without further purification. Solution of 1×10^{-3}mol L^{-1} omeprazole was prepared by dissolving 0.0867 g omeprazole in 20 mL twice-distilled water and then dissoluting to 250 mL. All of the chemicals were of reagent grade (Merck, Darmstadt). Twice-distilled deionized water served as a solvent.

Procedure: To evaluate the concentration of omeprazole in omeprazole enteric coated tablets, the standard curve method was used in the experiment. Transfer of the stock solution needed for assay into a 50 mL standard flask, followed by the addition 10.0 mL 1.0 mol L^{-1} HAc-NaAc buffer solution (pH 5.10) and made up to volume with distilled water. The solution was transferred into the electrolytic cell, then the pre-concentration step was performed in a stirred (ca. 500 rev min^{-1}) solution for 120 sec During this period, the glass carbon electrode was held at 0.40 V. The stirring was then stopped and after 10 sec the voltamperogram was recorded by applying the Differential Pulse Voltammetry (DPV) from 0.40 to 1.0 V and measured the peak height at about +0.74 V.

RESULTS

Regression line and detection limit: Under the optimum conditions and over a concentration range of 1.0~20 mg L^{-1} for omeprazole, the DPV peak height varied linearly with concentration of omeprazole and the equation of the regression line obtained was expressed as ip (μA) = 1.1062×c (mg L^{-1})+4.3012 ($n = 6$, $r^2 = 0.986$), the detection limit was 0.19 mg L^{-1}(S/N = 3).

Analysis of real sample: Dissolved the sample omeprazole enteric coated tablets, which was purchased from market and nominal 0.02 g per tablet, in the water and diluted to the volume 20 mL. Transferred of the solution needed for assayed into the electrolytic cell, the concentration of omeprazole was determined using the method of standard additions according to the voltammetric method described above and the results were shown in Table 1.

Table 1: Determination results of omeprazole in sample

No. of sample	Nominal (mg)	Determined (mg)	Added (mg)	Total (mg)	Recovery (%)
			10	30.6	102
05037	20	20.4	20	40.4	100
			30	50.2	99.3

DISCUSSION

The concentration, pH value and the type of buffer were important parameters that greatly influence the voltammetric behaviors of omeprazole. In order to achieve the maximum sensitivity of the omeprazole, different supporting electrolytes such as hydrochloric acid, potassium chloride solution, sodium hydroxide solution, Britton-Robinson buffer solution, HAc-NaAc buffer solution and ammonia/ammonium chloride buffer solution, were compared and the results showed that there was a oxidation peak in neutral or acid solution and the 0.20 mol L^{-1} HAc-NaAc solution was found to be best, the voltrammograms of omeprazole being well defined and the sensitivity reasonably high (Fig. 2).

When the initial potential less than 0.40 V, the peak height decreased with the decreasing potential. Within the chosen range of 0.30 to 0.50 V, the peak height kept stable, so 0.40 V was chosen as the initial potential. There was no effective affection on the peak height of the concentration of omeprazole when it was above 80 mg L^{-1}. The peak height increased with the pre-concentration time firstly, but reached stable after pre-concentration at 0.40 V for 120 sec if the concentration less than 80 mg L^{-1}, so pre-concentration for 120 sec and quite for 10 sec were selected in all these experiments.

The effects of several types of interfering species on the determination of 5.0 mg L^{-1} omeprazole were examined. The relative error range was below ±5% in the presence of 1000-fold sodium chloride, ammonium chloride, oxalic acid, citrate acid, tartaric acid, glucose, starch, or 100-fold histidine, glycine, glutamic acid, proline and 50-foldmethionine, tryptophan.

The typical repetitive cyclic voltammetric curves were shown in Fig. 3. An oxidation peak was observed in ~0.84 V and the oxidation peak in the first scan after an accumulation time of 120 sec which was much longer than in the second scan. No peak was observed in the catholic branch, indicating irreversibility of the oxidation.

The effect of the deposition time on the oxidation peak height of linear scan voltammetry was examined. The peak height increased with the adsorption time in the form of the adsorption isotherm. At relatively longer adsorption times, an equilibrium surface concentration was reached and the peak height became almost constant. Pre-concentration time of 120 sec the peak height, which varied linearly with concentration of the investigated compound showed the process, was diffusion controlled (Zhen *et al*., 1997).

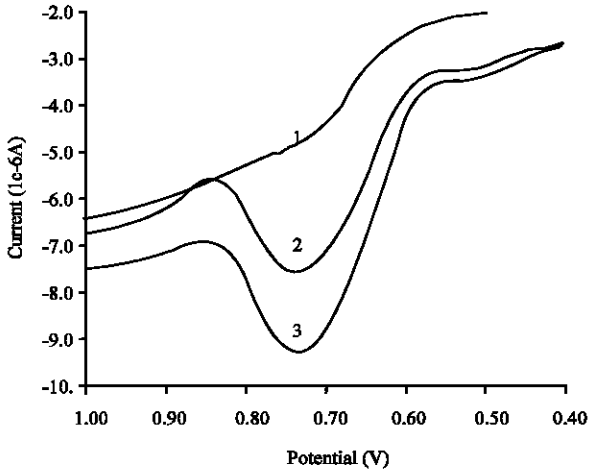

Fig. 2: Differential pulse voltammograms of omeprazole. Scan potential: 0.40 to 1.0V; pre-concentration time: 120 sec; quite time: 10 sec; scan rate: 50 mV sec^{-1}; pulse amplitude: 60 mV. 1, 0.2 mol/LHAc-NaAc (pH 5.10); 2, 3.0 mg L^{-1} omeprazole+0.2 mol/LHAc-NaAc (pH 5.10); 3, 5.0 mg L^{-1} omeprazole+ 0.2 mol/LHAc-NaAc (pH 5.10)

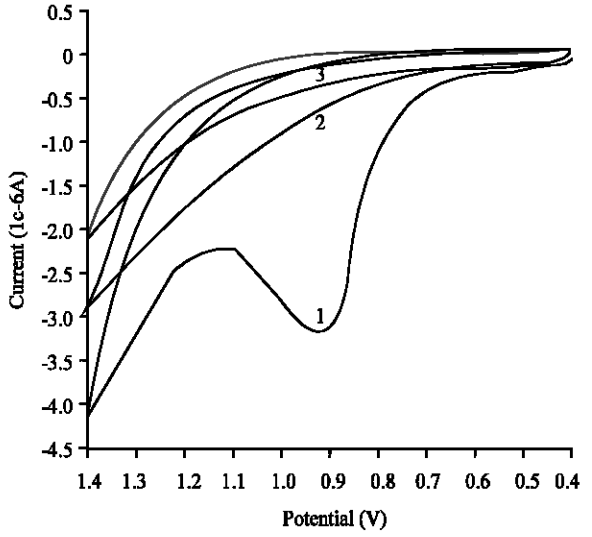

Fig. 3: Repetitive cyclic voltammogram of omeprazole 5.0 mg L^{-1} omeprazole + 0.2 mol/LHAc-NaAc (pH 5.10). Scan potential: 0.40 V to 1.0 V; pre-concentration time: 120 sec; Quite time: 10 sec; scan rate: 100 mV sec^{-1}. 1, first scan; 2, sec scan; 3, third scan

CONCLUSIONS

The utility of electrochemical analysis method using glass carbon electrode as working electrode for the determination of omeprazole was reported for the first time. In the medium of Hac-NaAc buffer solution (pH 5.10), a sensitive differential pulse voltammetric peak of omeprazole at glass carbon electrode at about +0.74 V (vs Ag/AgCl) is found. There is a good linear relationship between the peak current and the concentration of omeprazole in the range of 1.0~20 mg L^{-1}. The detection limit of the method is 0.19 mg L^{-1}. The electrochemical analysis method described here enables simple and rapid determination of omeprazole in real samples. The concentration of omeprazole in omeprazole enteric coated tablets has been determined with good results by this method.

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