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Square wave anodic stripping voltammetric determination of metoclopramide in tablet and urine at carbon paste electrode

O.A. Farghaly*, M.A. Taher[1], A.H. Naggar, A.Y. El-Sayed

Chemistry Department, Faculty of Science, Al-Azhar University, Assiut Branch, 71524 Assiut, Egypt

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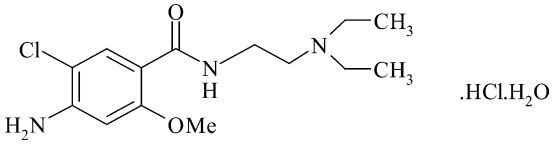
Abstract

A simple, reliable and selective square wave anodic stripping (SWAS) voltammetric method at carbon paste electrode (CPE) of metoclopramide hydrochloride (MCP) in pharmaceutical dosage forms (tablet) and in biological fluids (spiked and real urine samples) has been developed and evaluated. Different parameters such as medium, supporting electrolyte, pH, accumulation potential, scan rate, accumulation time and ionic strength, were tested to optimize the conditions for the determination of MCP. The adsorbed form is oxidized irreversibly under optimal conditions, viz., 0.4 M HCl–sodium acetate buffer (pH ~ 6.2), 0.2 M KCl, a linear concentration ranges from 0.067 to 0.336, 0.067 to 0.269 and 0.067 to 0.269 ng/mL of MCP, at accumulation times 60, 120 and 180 s, respectively, can be determined successfully. The interferences of some common excipients and some metal ions were studied. The standard addition method was used to determine the MCP in pure solutions, tablets and in biological fluids with satisfactory results. The data obtained are compared with the standard official method.

Keywords: Square wave voltammetry; Carbon paste electrode; Metoclopramide hydrochloride; Tablet; Urine

1. Introduction

Metoclopramide hydrochloride (MCP) is used as an antiemetic in the treatment of some forms of nausea and vomiting and to increase gastrointestinal motility. A study describing the pharmacokinetics and disposition of MCP in human's shows that the drug is rapidly and well-absorbed following oral administration with peak time at about 1 h. However, the bioavailability of MCP is quite variable and this arises from considerable inter-individual variation in metabolism [1]. The difference in the bioavailability of oral MCP in different subjects has potential clinical importance since it is anti-emetic effect and adverse effects on central nervous system correlate with the plasma concentration. Therefore, the measurements of MCP plasma concentration are essential to optimize therapy and avoid toxic concentrations. However, the structure of MCP is mentioned as below:



Many analytical methods have been developed for the analysis of MCP, most based on spectrophotometric [2–13], flourometric [14–16], ^{1}H NMR spectroscopic [17], chromatographic techniques [18–28], potentiometric [29–31], capillary electro phoresis [32] and radio immunoassay [33]. Most of these methods are complicated and need sophisticated instrumentation such as HPLC or spectrophotometry.

Stripping voltammetry is a very sensitive method for the determination of many traces of organic compounds and metal ions achieving it is low level of detection by combining an accumulation process with a voltage scanning measurements [34–38]. Carbon paste electrodes are convenient

* Corresponding author. Present address: Al-Azhar University, Faculty of Science, Chemistry Department, 71524 Assiut, Egypt.
Tel.: +20 88 312193; fax: +20 88 325436.
E-mail address: othman15@yahoo.com (O.A. Farghaly).
[1] Present address: Faculty of S. Society, Baha, Om El-Kora University, Saudi Arabia.

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and often used as working electrodes for the voltammetric measurements because of their attractive properties. From analytical point of view, these electrodes exhibit rather low background currents over a wide range of potentials when compared with other solid electrodes, and after a renewability of their surface as well as a high versatility and simplicity of modification [35,39].

The present work is a continuation of our studies in the field of drug analysis using mercury and modified carbon paste electrodes [36–40]. At present only one study dealing with MCP electrochemical oxidation behavior have been reported [41]. The voltammetric determination of MCP by SWASV at a paraffin oil bare carbon paste electrode has not been studied yet.

Thus, the aim was to investigate the square wave anodic stripping voltammetric determination of MCP in dosage forms (tablets) and in biological fluids (spiked and real urine sample) at a paraffin oil bare carbon paste electrode (CPE).

2. Experimental

2.1. Apparatus

All voltammetric experiments were performed with EG&G Princeton Applied Research (PAR Princeton, NJ, USA) Model 273 A potentiostat, controlled by the Model 270/250 electrochemical software version 4.30. A three-electrode cell was employed incorporating a hand-make working carbon paste electrode that prepared as previously mentioned [35], an Ag/AgCl (saturated KCl) reference electrode and a platinum wire was used as a counter electrode. Mass transport was achieved with a Teflon-coated bar at approximately 400 rpm using a magnetic stirrer (KIKA Labortechinik, Germany). All the pH measurements were made with VWR Scientific Products Model 2000.

2.2. Reagents and materials

All of the chemicals used were of analytical grade (Merck and Sigma), and all of the solutions were freshly prepared in doubly distilled water.

Metoclopramide hydrochloride (MCP) (Merck) stock standard aqueous solution (1×10^{-2} M) was prepared (at 25 C° 48 g MCP per 100 mL doubly distilled water [42]) and kept in a brown volumetric flask. MCP working standard solutions were prepared daily by serial dilution of the stock standard solution.

Pharmaceutical formulations: Primperan® tablet (Memphis Co. for Pharm. & Chem. Ind. Cairo, A.R.E.) labeled to contain 10 mg MCP per tablet.

2.3. General analytical procedure

The preconcentration step was performed by immersing the carbon paste electrode in a stirred 10 mL sample solution for a given period of time at potential range from +0.45 to +1.3 V. The stirring was then stopped and after a delay period of 10 s to settle the solution and decrease the background current, cyclic or square wave voltammogram was recorded in the positive potential direction. A renewed carbon paste surface was used for each measurement.

Table 1
The optimum operational parameters selected for the determination of MCP by SWASV at CPE

Parameter	Selected value
Accumulation potential	+0.45 V
Final potential	+1.3 V
Modulation time	10 s
Frequency	50 Hz
Scan increment	2 mV
Accumulation time	Various
pH	6.2
Buffer type	0.4 M HCl–sodium acetate buffer
Supporting electrolyte	0.2 M KCl

For determination of MCP in biological fluids (spiked and real urine samples), 1 mL aliquot of urine (blank or containing drug) was transferred to a 250 mL separating funnel containing 5 mL of diethyl ether (Merck). The mixture was thoroughly shaken for 15 min, then, the organic layer was transferred to a glass tube, and the solvent was evaporated in water bath to dryness. The residue was reconstituted in doubly distilled water. Then, 20 μL urine sample (containing 6.73 ng/mL of the drug in case of spiked urine and unknown a mount of excreted drug in real urine samples), was added to 10 mL voltammetric cell and mixed thoroughly with 0.4 M HCl–sodium acetate buffer pH ∼ 6.2 containing 0.2 M KCl. The solution was stirred at 400 rpm at open circuit conditions and the square wave voltammogram was recorded. Also, in case of dosage forms, 10 tablets of the drug were weighed into a small dish, powdered and mixed well. A portion equivalent 0.1294 g was weighed and dissolved in 100 mL of doubly distilled water, shaken well and filtered using filter paper. An aliquot of the filtrate was then transferred into a calibrated flask and it was completed to volume with the same solvent. 20 μL of each solution was then added to the measurement cell. In all measurements, the square wave voltammogram was recorded in positive potential direction. Table 1 contains the optimum operational parameters selected for the determination of MCP by SWASV using CPE.

3. Result and discussion

3.1. Cyclic voltammetry

The cyclic voltammogram of the oxidation of metoclopramide hydrochloride at paraffin oil bare carbon paste electrode in HCl–sodium acetate buffer (pH ∼ 6.2) containing 0.2 M KCl was studied. In the forward scan, one well-defined anodic peak owing to the oxidation of amino group was observed and no peak was noticed in the reverse direction. The

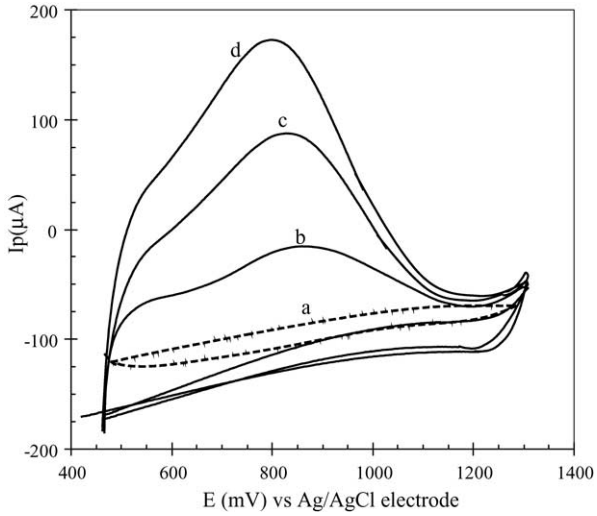

Fig. 1. Cyclic voltammogram of 33.63 μg/mL MCP at 60 s accumulation time, using 0.4 M HCl–sodium acetate buffer (pH ~ 6.2), containing 0.2 M KCl, accumulation potential +0.45 V, CPE 15% pasting oil, at different scan rates: (a) residual current, (b) 50 mV/s, (c) 75 mV/s and (d) 100 mV/s scan rate.

peak current decrease with succeeding potential scans suggesting an adsorbed species formation on the electrode surface. This indicates that the oxidation of MCP is irreversible [41]. The effect of scan rate, ν, on the peak current and peak potential was evaluated and showing in Fig. 1. The peak potential was shifted to less positive values on increasing scan rate, which confirms the irreversible nature of the oxidation process.

3.2. Effect of accumulation potential

The effect of accumulation potential on the peak current was also investigated in potential range from +0.25 to +0.8 V at 10 s preconcentration time for 13.45 μg/mL MCP solution (pH ~ 6.2) as showed in Fig. 2. Experiments proved that the peak current of MCP increases with negative shifting of starting potential in the range from +0.8 to +0.45 V and then decrease with negative shifting from +0.45 to +0.25 V. The Peak current has its maximum value at initial potential +0.45 V, which was used in the subsequent examinations of other decencies.

3.3. Effect of buffer type, pH, supporting electrolyte and ionic strength

The effect of type of buffer used as electrolyte (acetate buffer, citrate buffer, phosphate buffer, Britton–Robinson and HCl–sodium acetate buffer) on the analytical signal was tested. Both the peak height and peak shape were taken into consideration when choosing type of buffer. A study of the influence of the ionic strength of the medium on the definition of the voltammetric peak revealed that minimal background current, the best curve and the highest peak were obtained in 0.4 M HCl–sodium acetate buffer.

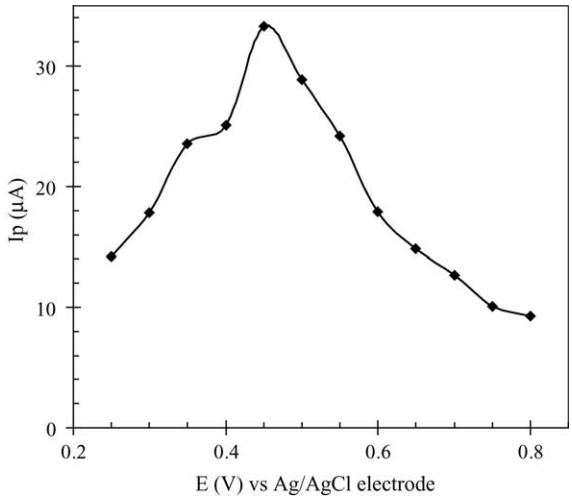

Fig. 2. Effect of accumulation potential on the peak current of 13.45 μg/mL MCP using 0.4 M HCl–sodium acetate buffer (pH ~ 6.2), at 10 s, CPE 15% pasting oil.

The effect of pH on the oxidation of MCP at CPEs was studied over the pH range 2.5–11.2 at the concentration 13.45 μg/mL MCP by square wave voltammetry as shown in Fig. 3. A small current was observed at pH ~ 3.5, which increased gradually up to pH ~ 6.2 and then decreased at higher pH. Thus, pH ~ 6.2 was used in all measurements. The potential of anodic peak of MCP is shifted linearly towards less positive values with increasing the pH over than 6.2.

The influence of type of supporting electrolyte (KCl, $NaNO_3$, $NaClO_4$) was studied. The best choice that has ability to give the best shape and highest current was KCl. The addition of 0.1 M KCl to the voltammetric cell contain HCl–sodium acetate buffer (pH ~ 6.2) containing

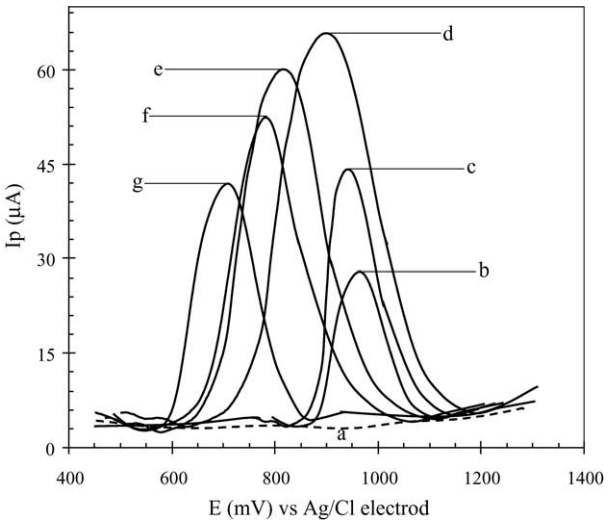

Fig. 3. Typical SWAS voltammogram for effect of pH on 13.45 μg/mL MCP at +0.45 V, using 0.4 M HCl–sodium acetate buffer, containing 0.2 M of KCl: (a) residual current, (b) pH ~ 2.54, (c) pH ~ 4.55, (d) pH ~ 6.20, (e) pH ~ 7.94, (f) pH ~ 9.36 and (g) pH ~ 11.11.

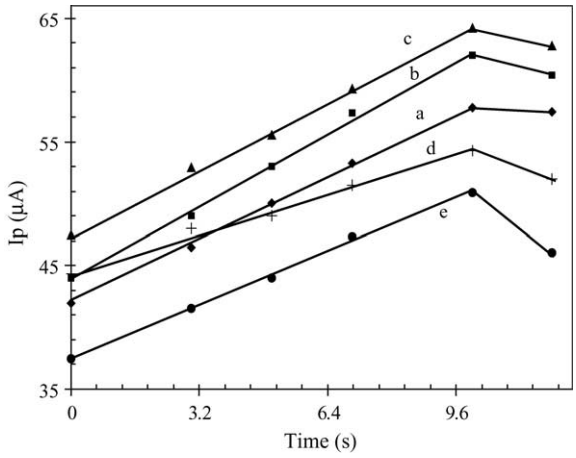

Fig. 4. Plot of current versus accumulation time of 13.45 μg/mL MCP using 0.4 M HCl–sodium acetate buffer in presence of different concentrations of KCl: (a) 0.05 M, (b) 0.1 M, (c) 0.2 M, (d) 0.3 M and (e) 0.4 M of KCl.

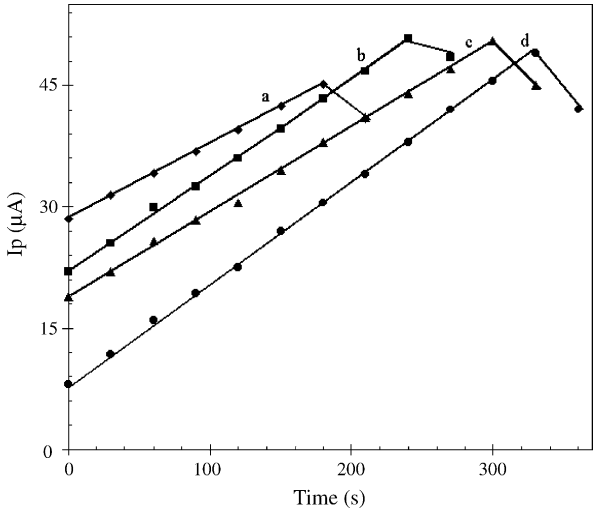

Fig. 5. Current–time plots at different concentrations of MCP, using 0.4 M HCl–sodium acetate buffer, containing 0.2 M of KCl: (a) 67.26 ng/mL, (b) 6.726 ng/mL, (c) 0.6726 ng/mL and (d) 0.06726 ng/mL of MCP.

13.45 μg/mL MCP at 10 s accumulation time cause large excess in peak current when compared with the similar peak current in absence of KCl.

The influence of ionic strength on the efficiency of accumulation of 13.45 μg/mL MCP was studied for a 10 s preconcentration time. Changing the KCl concentration in range from 0.05 to 0.4 M in the chosen buffer type varied the ionic strength. The result showed that increasing ionic strengths were found to be of great significance on the degree of accumulation. This indicated that the process responsible for accumulation of the drug at the electrode surface was mainly electrostatic in nature. The effect of concentration of KCl was very important as can be seen in Fig. 4. However, the best accumulation is attained in presence of 0.2 M KCl.

3.4. Effect of accumulation time and reproducibility

The dependence of the peak current on accumulation time was studied for four levels of concentration named as: 0.067, 0.673, 6.735, 67.260 ng/mL MCP. The stripping signal increased linearly with increase accumulation time up to 330, 300, 240 and 180 s for 0.067, 0.673, 6.735, 67.260 ng/mL MCP, respectively (Fig. 5). Repeating three experiments on 0.673 ng/mL MCP at 60 s accumulation time checked the reproducibility of the adsorption process. The relative standard deviation was computed to be 4%.

3.5. Effect of concentration and detection limit

The square wave anodic stripping peak for MCP yields a well-defined concentration dependence using SWASV method. The calibration plots over the MCP concentration range, following different preconcentration times were investigated. However, a well defined peak was observed over the concentration range 0.067–1.682 ng/mL MCP at 60, 120 and 180 s (Fig. 6) and over the range from 2.354 to 104.589 ng/mL MCP, after 60, 120 and 180 s, respectively, with the stirring at +0.45 V. The results show positive deviation from linearity at concentrations higher than 0.336 ng/mL at 60 s and 0.269 ng/mL at 120 and 180 s, and over the concentrations 37.329, 30.603 and 17.151 ng/mL of MCP at 60, 120 and 180 s, respectively. However, Table 2 illustrates the linearity ranges.

The detection limits estimated [35,39,43] as $3\sigma/b$, where b is the slope and σ = standard deviation (S.D.) of the intercept and the quantitative limits also calculated as $10\sigma/b$. The results obtained from the proposed method shown that MCP can be detected from 2×10^{-11} M (0.007 ng/mL), with standard deviation 0.51%, correlation coefficient $r = 0.9985$ ($n = 5$) at accumulation time 60 s. These results were compared with two reported methods in which MCP was determined. In the first method MCP was determined by second-derivative adsorptive anodic stripping voltammetry with a Nafion modified glassy carbon electrode with detection limit

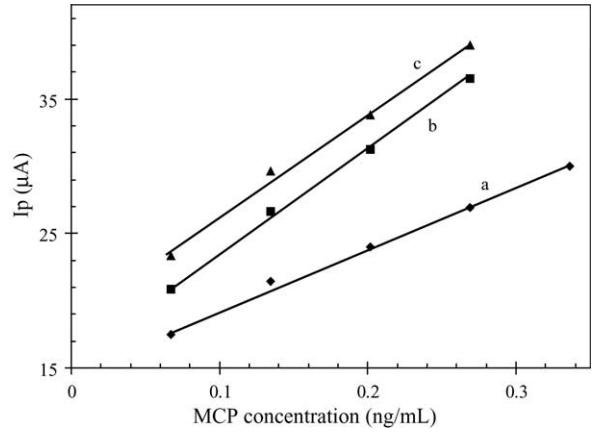

Fig. 6. Plot of I_p vs. concentrations of MCP using 0.4 M HCl–sodium acetate buffer (pH ~ 6.2), containing 0.2 M of KCl at different accumulation times: (a) 60 s, (b) 120 s and (c) 180 s.

Table 2
Characteristic of linear regression of calibration curves for MCP in 0.4 M HCl–sodium acetate buffer (pH ~ 6.2) using SWASV at different deposition times

Deposition time (s)	Linearity range (ng/mL)	Correlation coefficient	Slope (μA/(ng mL)) ± S.D.	Intercept (μA) ± S.D.
60	0.067–0.336	0.9953	49.821 ± 2.787	14.215 ± 0.621
	2.354–37.329	0.9971	0.745 ± 0.023	24.602 ± 0.478
120	0.067–0.269	0.9921	67.335 ± 6.249	16.960 ± 1.151
	2.354–30.603	0.9977	0.866 ± 0.026	26.489 ± 0.441
180	0.067–0.269	0.9933	73.624 ± 6.055	19.040 ± 1.115
	2.354–17.151	0.9931	0.807 ± 0.055	30.204 ± 0.514

at 0.027 ng/mL, and 4 min accumulation time [41]. The second one suggest that MCP was determined spectrophotometrically. In this method there is a complex formed upon the oxidation of MCP with Fe^{3+} in presence of *o*-phenanthroline or bibyridyl, with the detection limit at 70 ng/mL of the drug [2]. As a result of this comparison, the proposed method was very sensitive than the reported methods. The repeatability of the peak current at new surfaces as measured by relative standard deviation (R.S.D.) was 3.2% and at the same electrode after consecutive accumulation and cleaning step (the paste was cleaned by washing with doubly distilled water) was 3.6% ($n = 3$).

3.6. Effect of interferences

To test the efficiency and selectivity of the proposed analytical method to pharmaceutical formulations, a systematic study of sample solutions containing a fixed amount of MCP (67.260 ng/mL) spiked with excess amount of some common excipients and additives that are used in pharmaceutical preparations **(**10 000:1**)** (e.g. glucose, sucrose, lactose and fructose) under the optimum experimental conditions was made to know the effect of such excipients and additives on the efficiency and selectivity of the proposed analytical method to pharmaceutical formulations.

Experimentals showed that there was no serious interference occurred from the classical additives tested. Therefore, the proposed method can be used as a selective method. The influence of ascorbic acid, which is a potentially interfering compound present in biological samples, was investigated. It was found that an equimolar concentration or even at higher molar excess (10 000:1) of ascorbic acid had no effect on the peak response of MCP. Also interference of some metal ions was tested under the same conditions, It was observed that 10 000-fold excess of Al(III), Ca(II), Sr(II), Cd(II), Ni(II), Co(II), Mg(II), and Cu(II) metal ions had no effects on MCP determination. However, Table 3 shows the results for such additives.

Table 3
Tolerance of the proposed method to interference

Additives	Tolerance molar ratio (M/M)
Ascorbic acid	10000
Metal ions {Al(III), Ca(II), Sr(II), Cd(II), Co(II), Ni(II), Mg(II) and Cu(II)}	10000
Glucose, sucrose, lactose, fructose	10000

3.7. Analytical applications

The proposed method was successfully applied to determine MCP in pharmaceutical preparations, spiked and real urine samples.

3.7.1. Pharmaceutical preparations

The square wave voltammogram of the used tablet sample was recorded after preconcentration time for 30 and 60 s, in 0.4 M HCl–sodium acetate buffer (pH ~ 6.2) containing 0.2 M KCl solution. The content of the tablet in the cell was determined by standard addition method [44]. One peak was observed on addition of pure drug to the sample at +0.9 V (Fig. 7). On increasing the MCP concentration, the peak current was increased linearly from 20.178 to 302.670 ng/mL at 30 s and from 20.178 to 168.150 ppb at 60 s which fitted the equation $Y = 0.075X + 43.015$ with correlation coefficient of 0.9967 and $Y = 0.095X + 48.536$ with correlation coefficient 0.9934, respectively. The determination of MCP in Primperan® tablets at the lower accumulation time (30 s) may be owing to the absence of chloride in these tablets

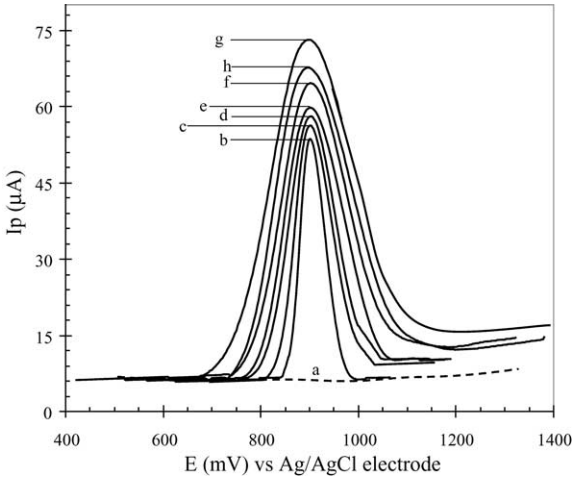

Fig. 7. Typical SWAS voltammogram of Primperan® tablet in presence of 0.4 M HCl–sodium acetate buffer (pH ~ 6.2), containing 0.2 M of KCl at 60 s: (a) residual current, (b) sample solution, (c) 20.178 ng/mL, (d) 26.904 ng/mL, (e) 33.63 ng/mL, (f) 100.89 ng/mL, (g) 168.15 ng/mL and (h) 235.41 ng/mL MCP.

Table 4
Analysis of MCP in tablet, spiked and real urine sample

Sample	Accumulation time (s)	Detection limits (ng/mL)	Linearity range (ng/mL)	Slope (μA/(ng mL)) ± S.D.	Intercept (ng/mL) ± S.D.	Correlation coefficient
Primperan® tablet[a]	30	15.320	20.178–302.670	0.075 ± 0.003	43.015 ± 0.383	0.9967
	60	14.368	20.178–168.150	0.095 ± 0.006	48.536 ± 0.455	0.9934
Spiked urine sample	60	0.111	0.201–3.026	10.548 ± 0.239	28.950 ± 0.392	0.9987
	120	0.131	0.201–2.354	11.764 ± 0.406	33.943 ± 0.514	0.9976
	180	0.127	0.201–1.681	14.403 ± 0.675	38.370 ± 0.609	0.9967
Real urine sample after 12 h	30	18.400	26.904–1042.53	0.030 ± 0.001	34.974 ± 0.184	0.9992
	60	20.250	26.904–369.930	0.048 ± 0.002	43.908 ± 0.324	0.9966
Real urine sample after 24 h	60	0.733	0.673–16.815	1.064 ± 0.036	49.452 ± 0.260	0.9965
	120	0.327	0.673–3.363	1.217 ± 0.065	58.461 ± 0.133	0.9942

[a] The *t*-test (1.43) and *f*-test (0.1) values were less than the theoretical values of *t*- and *f*-values (2.6 and 0.15) at 95% confidence limit for five degree of freedom.

(MCP base), where excess of chloride may be cause increase the acidity of medium which higher residual current and in turn low sensitivity were obtained in synthetic solution [46]. The obtained values were compared statistically by Student's *t*-test for accuracy and *f*-test for precision with the official method [42,45] (depending on potentiometric method using 0.10 M perchloric acid) at the 95% confidence level with five degrees of freedom, as recorded in Table 4. The result showed that the *t*- and *f*-test values were less than the critical value, indicating that there was no significant difference between the proposed and the official methods. Because the proposed method was more reproducible with high recoveries than the official method, it can be recommended for the routine analysis in the majority of drug quality control laboratories.

3.7.2. Spiked urine samples

The proposed method was applied to the determination of MCP in spiked urine samples from healthy volunteers using standard addition method. Fig. 8 showed the square wave response to definite concentration of MCP in urine samples, after 60 s accumulation time of MCP. The electrode response was linearly related to the MCP concentration within the range 0.201–3.026 ng/mL of MCP, which applied to line with equation $Y = 10.548X + 28.950$ with correlation coefficients of 0.9987; standard deviation for slope and intercept of the calibration curve were 0.239 and 0.392%, respectively. The detection limit was 0.111 ng/mL at 60 s accumulation time (S.D. = 0.392% and $r = 0.9998$ ($n = 5$)). The repeatability of total analytical process was determined from multiple measurements at each of the urine samples ($n = 5$). An average deviation of 3.5% was obtained.

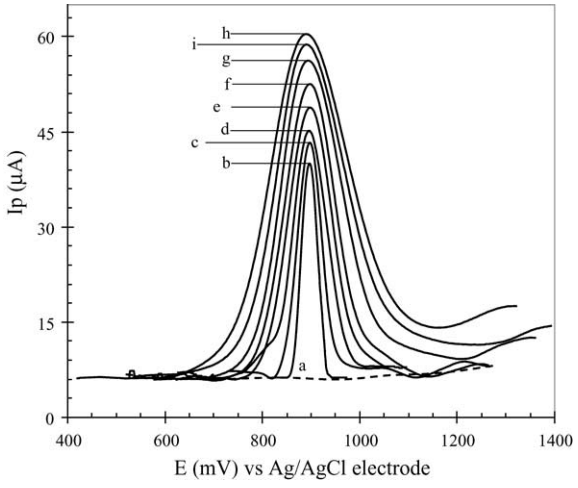

Fig. 8. Typical SWAS voltammograms of MCP in spiked urine sample at 60 s accumulation time in 0.4 M HCl–sodium acetate buffer containing 0.2 M KCl: (a) residual current, (b) 6.73 ng/mL, (c) 6.93178 ng/mL, (d) 7.20082 ng/mL, (e) 7.53712 ng/mL, (f) 8.54602 ng/mL, (g) 10.22752 ng/mL, (h) 12.58162 ng/mL and (i) 15.60832 ng/mL of MCP.

3.7.3. Real urine samples

The proposed method was also applied to the determination of MCP in human urine samples from healthy volunteers who received a single oral dose of 10 mg of Primperan® tablet. The samples of individuals were collected for up to 24 h after administration of Primperan® and urinary volumes were recorded as well. MCP was well separated from organic components and excipients did not interfered [35]. The result obtained summarized in Table 4, shown that a small amount of an administered dose are excreted in the urine. The results showed a high correlation coefficient ($r \geq 0.9992$). Also, the obtained result from the proposed method for voltammetric assay of MCP in real urine samples were compared with those obtained by official method [42,45], in which about 60% of an oral dose is excreted in the human urine in the first 24 h.

3.8. Accuracy and repeatability

Applying the proposed method for the analysis of dosage forms and urine validated the accuracy of the suggested procedure. The analysis of MCP in spiked and real urine samples exhibited the correlation coefficient of 0.9965, the standard deviation of both slopes of ≤0.57% and the intercept of ≤0.89%, indicating adequate precision and accuracy of the proposed method.

4. Conclusion

The SWASV method with carbon paste electrode for the quantitative determination of MCP was found to be simple and highly sensitive. A detection limit of 2×10^{-11} M (0.007 ng/mL) at 60 s accumulation time with the standard deviation 0.51% was obtained in pure solutions. The method can be used successfully to assay the drug in dosage form as well as in spiked and real urine samples.

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