ENGLISH SUMMARY

Summary

This work comprises of three chapters

Chapter I: Includes the literature survey on the pervious electrochemical and analytical studies that has been made on the pharmaceutical compounds under investigation.

Chapter II: This chapter includes the experimental part. It describes the materials, preparation of samples and solutions for different used technique, also the instruments used in this study.

Chapter III: It includes the results and discussions of the resultes obtained from the research study on the compounds under consideration. It comprises five parts:

1. Ciprofloxacin hydrochloride

Ciprofloxacin hydrochloride is broad spectrum antibiotic. The electrochemical behaviour of this compound is studied in different pH values using DC-polarography. One polarographic wave appears in the pH range 2.0-11.5. The half-wave potential $E_{1/2}$ is pH dependent, being shifted to more negative values with increasing of pH value. The wave height i_l is slightly decreases with increasing of pH, this may be due to the increasing of the viscosity of the medium. The logarithmic analysis of the polarographic waves indicates that the reduction process proceeds irreversibly. The effect of mercury height on i_l indicates that the electrode process is mainly controlled by diffusion with adsorption contribution. The number of protons (Z_H^+) and electrons (n_a) involves in the rate determining step is found to be one proton and two electrons, respectively. Ciprofloxacin hydrochloride forms (1: 2) complexes with Cd (II) and with Zn(II) on using DC-polarography in 0.1 M sodium perchlorate.

The cyclic voltammetric behaviour of ciprofloxacin hydrochloride investigated at glassy–carbon electrode surface in B.R buffer solutions of pH 3.6, 7.0 and 10.0 at different scan rates 20-500 mV/s. One cathodic peak appeared. The peak potential E_p is shifted to more negative value with rise of pH. The plot of E_p versus ln υ gives linear correlations with slope values proportional to (αn_a). Plotting i_p versus square root of the scan rate gives linear correlations deviating from the origin conforming that the electrode process of ciprofloxacin hydrochloride is controlled by diffusion with some adsorption contribution.

Accumulation of ciprofloxacin hydrochloride at the glassy-carbon electrode using Cathodic adsorption Square Wave Stripping Voltammetry (CAdSWSV) is found to be optimized in B.R. buffer of pH 4.2 under the experimental and instrumental conditions: deposition potential E_d is -100 mV, accumulation time t_d is 300 sec, scan rate is 1.0 mV/s, pulse width is 200 mV and pulse height is 200 mV. The method is applied successfully for analysis of dosage forms such as Ciprofloxacin and Rancif tablets. The detection limit is 2.0×10^{-9} M (7.7×10^{-4} µg/ml).

Spectrophotometric behaviour of ciprofloxacin hydrochloride is studied using three analytical reagents Sudan II (I), Congo-red (II) and gentian violet (III). The optimum pH values at which the well developed band represent the complex formation are reported to be 5.5, 2.5 and 2.5 for I, II and III respectively. The maximum wavelength at which the complexes are formed are 550, 517 and 585 nm with reagents I, II and III, respectively. Effect of time and temperature are studied, the complexes are formed at once at room temperature and remain stable for 24 h. The ratio of the formed complexes are studied by two methods; molar ratio and

continuous variation and it is found to be (1:1) with all reagents. The effect of reagent concentration is also studied, the most suitable concentration is 1 ml of 1 x 10^{-3} M for all reagents. Beer's law limits are calculated at optimum condition and are confirmed by using Ringbom method. The study of the accuracy of the proposed method is done by using F-test and t-test. The proposed method applied to tablet form of the drug shows high accuracy in comparison with the official method.

2. ofloxacin

Ofloxacin is considered to be broad spectrum antibiotic. DC-polarographic behaviour of 2 x 10^{-4} M ofloxacin is studied in B.R buffer solution of pH ranging from 3.5 to 10.5 containing 20 % (v/v) ethanol. Only one polarographic wave is appeared, this wave due to the reduction of the carbonyl group. Analysis of this polarographic wave indicates that the reduction process proceeds irreversibly and it is mainly controlled by diffusion with some adsorption contribution. The rate determining step involves one proton and two electrons. Cyclic voltammetric behaviour of ofloxacin at the glassy carbon electrode surface in pH 3.4, 7.0 and 10.0 at different scan rates confirmes the previous behaviour of the electrode process. Ofloxacin formes 1:2 complexe with Cd(II) and Zn(II) using DC-polarography in 0.1 M sodium perchlorate.

Determination of ofloxacin using CAdSWSV is performed at the glassy-carbon electrode after optimizing both in B.R. buffer solution of pH 4.2 at E_d = -200 mV, frequency = 5000 Hz, t_d = 300 sec, scan rate is 10 mV/s, pulse width is 100 mV and pulse height is 100 mV. The method is applied successfully for analysis of dosage forms. The detection limit is 1 x 10^{-10} M (2.2 x 10^{-4} µg/ml).

Spectrophotometric method is used for determining ofloxacin using regents under investigation. Different experimental conditions are optimized; at pH 6.0, 8.5 and 11.0 on using I, II and III respectively, the reagent concentration is 1 ml of 1 x 10^{-3} M for all reagents. Experiments show that the complexes are formed spontaneously at room temperature. The ratio of the formed complexes is 1:1 with all reagents. Beer's law limits are detected. Statistical studies show high accuracy of the proposed method in comparing with the official method for determination of ofloxacin in pure and in tablet forms.

3- Norfloxacin:

One polarographic wave corresponding to the reduction of the carbonyl group appeared on following of the reduction behaviour of norfloxacin in B.R. buffer solution of pH from 3.2 to 10.9 containg 20 % (v/v) ethanol. Cyclic voltammetric behaviour of 1 x 10^{-4} M of norfloxacin is investigated in B.R. buffer solution of pH values 4.6, 7.4 and 10.4 containing 20 % (v/v) ethanol at different scan rates from 20 to 500 mV/s. The study for both techniques shows that the electrode process is irreversible and controlled mainly by diffusion with some adsorption contribution. Complexation of norfloxacin with some metal ions is studied using DC-polarography in 0.1 M sodium perchlorate it is found that it forms 1:2 complexes with both Cd(II) and Zn(II).

CAdSWSV behaviour of 1 x 10^{-6} M norfloxacin is studied at the surface of glassy carbon electrode. A well defined peak is observed in B.R. buffer solution of pH 9.2, E_d = -500 mV, frequency = 5000 Hz, pulse width = 200 mV, pulse height = 200 mV, t_d = 300 sec and scan rate = 1.0 mV/s.

The method applied successfully for analysis of norfloxacin in its dosage forms. The detection limit is 2.5×10^{-9} M (7.98×10^{-4} µg/ml) .

Spectrophotometric behaviour of norfloxacin is studied in complexation with the analytical reagents under investegation. The complexes formed in optimum conditions pH values and wavelengths, also the effect of time and temperature are studied, the complexes are formed at once at room temperature. The ratio of complexes formed is found to be 1:1 with all reagents. Beer's law limits and Sandell sensitivity are calculated at the optimum conditions. The proposed method showed high accuracy in comparison with the official method.

4- Sildenafil citrate:

Reduction of the sexual stimulant sildenafil citrate occurred in B.R. buffer solution of pH from 3.5 to 10.0 containing 20% (v/v) ethanol using DC-polarographic technique. Only one wave is recorded. The analysis of the obtained wave shows that the electrode process is irreversible process and controlled mainly by diffusion with some adsorption contribution. Also reduction of sildenafil citrate is performed by using cyclic voltammetric technique at glassy–carbon electrode surface in three different pH at different scan rates. One cathodic peak is appeared, analysis of this peak confirmes the results obtaind by DC polarographic technique.

Determination of Sildenafil citrate in pure and tablet form was performed using CAdSWSV. The method is performed in B.R. buffer of pH 7.5 at E_d -400 mV, frequency 5000 Hz, t_d is 300 sec, scan rate is 10 mV/s, pulse width is 200 mV and pulse height is 200 mV. The detection limit is 3.7×10^{-9} M (1.65×10^{-3} µg/ml).

Spectrophotometric behaviour of sildenafil citrate is studied using reagents I, II and III. The experimental conditions for obtained well developed band represent the complex formation are reported. The ratio of the formed complexes is detected to be 1:1. Beer's law limits are calculated at optimum condition. Accuracy of the proposed method is high in comparing with the official method.

5- Chloramine-T:

DC-polarographic behaviour of the antiseptic agent chloramine-T is studied in B.R. of different pH values in the range 2.9-11.2 containing 20% (v/v) DMF. One polarographic wave is appeared. The limiting current i_l of the formed waves is nearly constant at pH $\leq$ 6.9 and decreases to its half value at pH > 6.9. This behaviour may be due to the formation of hardly reducible species in the alkaline medium. Polarographic parameters are calculated revealing that one proton and two electrons involved in the rate determining step. The irreversible electrode process is mainly controlled by diffusion with adsorption contribution. The cyclic voltammetric behaviour of chloramine-T is investigated in pH 3.3, 7.5 and 10.4 at different scan rates 20-500 mV/s. One cathodic peak is appeared. Analysis of this peak conformes that the same behaviour as that of DC-polarography.

CAdSWSV behaviour of chloramine-T in B.R. buffer of pH 7.2 at: E_d is -400 mV, t_d is 300 sec, scan rate is 1.0 mV/s, pulse width is 200 mV and pulse height is 200 mV is performed. The detection limit is 5.7×10^{-9} M (1.6×10^{-3} µg/ml).