



Study of Variation in Electrochemical Behavior of Metronidazole and Norfloxacin Simultaneously by Differential Pulse Voltammetry

V. M. Wagh*

Department of Chemistry, Ramnarain Ruia Autonomous College, Matunga (East),
Mumbai-400019.

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*Corresponding Author Email: vwagh123@gmail.com

Abstract

In present study, a successful attempt has been made to study the variation in electrochemical behavior of Metronidazole and Norfloxacin simultaneously using Differential Pulse Voltammetry (DPV) technique. The effect of different pH (2-10) of Britton-Robinson Buffer on voltammogram using 1M KCl as a supporting electrolyte was studied. The optimum pH was found to be pH 6.5. Both Metronidazole and Norfloxacin exhibited reduction cathodic peak at optimum pH with peak potential at -0.46 V for Metronidazole and -1.30 V for Norfloxacin vs. S.C.E. 0.1N CH₃COOH was used as Solvent for the analysis. The variation in electrochemical behavior of Metronidazole and Norfloxacin simultaneously at the optimized pH was studied by varying pulse amplitude and scan rate. The optimized pulse amplitude was found to be 50mV and the optimized scan rate was found to be 15 mV/s.

Keywords

Voltammetry, Metronidazole, Norfloxacin, Electrochemical.

INTRODUCTION

The main objective of study is to provide optimized parameters such as pH, pulse amplitude and scan rate of voltammogram for Metronidazole and Norfloxacin simultaneously which can be used in the method development and validation of Metronidazole and Norfloxacin in combined pharmaceutical formulations using Differential Pulse Voltammetry technique.

Individual determination of several drugs by various electroanalytical methods has been reported [1-4]. Simultaneous determination of drugs using conventional methods such as HPLC and spectroscopy have been reported [5-11]. For development and validation of any method based on voltammetric technique, the optimization of parameters is very important. The optimized parameters such as pH, pulse amplitude and scan rate can be useful in the simultaneous detection and determination of pharmaceutical formulation by voltammetric technique. It can also be used for devising electro-sensors for those pharmaceutical drugs.

Metronidazole, C₆H₉N₃O₃ that is 2-(2-methyl-5-nitro-1H-imidazol-1-yl)ethanol, is an antibiotic, amebicide, and antiprotozoal (Molecular weight: 171.15 g/mol) It is highly effective for bacterial and protozoan infections and is available in the tablet form.

Norfloxacin, $C_{16}H_{18}FN_3O_3$ that is (1-ethyl-6-fluoro-4-oxo-7-piperazin-1-yl-1H-quinoline-3-carboxylic acid) (Molecular weight: 319.331 g/mol)] is used in the treatment of bacterial infection.

MATERIALS AND METHODS:

Electrochemical Workstation



Electrochemical workstation- PG STAT 30 with 663 VA Electrode stand (Metrohm)

It is made up of three electrode system namely-

- 1) Hanging Mercury Drop electrode (HMDE) as the working electrode
- 2) Saturated calomel electrode as the reference electrode
- 3) Platinum electrode as the counter electrode

The pH measurements were made with Eupitrances model No. 610.

SOLUTION PREPARATION:

Combined stock solution of standard MZ and standard NF (1000 $\mu\text{g/mL}$ + 1000 $\mu\text{g/mL}$)

100mg of standard MZ and 100mg standard NF was accurately weighed and transferred into 100 mL standard flask, about 80 mL of 0.1N acetic acid was added to it. The mixture was sonicated for 10 minutes to dissolve the standards with intermittent shaking. The volume was made up to the 100 mL mark by adding 0.1N acetic acid.

Preparation of Britton-Robinson buffer:

0.204 g of boric acid, 2.8 mL of (85%) phosphoric acid and 2.3 mL of glacial acetic acid were transferred to three separate 100 mL of volumetric flasks and the volume of each flask was made up to the 100 mL mark with distilled water. These three solutions are then mixed in a beaker to get the solution of pH 1.8. The pH of the resulting solution was adjusted to the desired value by adding required quantity of 1M NaOH.

Preparation of the supporting electrolyte solution (1M KCl).

7.46 g of A.R. KCl were weighed and transferred into a 100 mL volumetric flask. About 80 mL of distilled water was added to dissolve the solid completely and then the volume was made up to the 100 mL mark with distilled water.

Optimization of the pH

The response of MZ and NF combination was studied over the pH range 2 to 10 in Britton – Robinson buffer. It was observed that peak shape and linearity (R^2) was best at pH = 6.5 for both MZ and NF, therefore pH = 6.5 was selected as the optimum pH for this combination.

• Effect of pH on polarogram of MZ and NF: -

Polarogram of MZ and NF combination were recorded at different pH (2-10) at fixed scan rate (15 mV/s) and at fixed pulse amplitude (50 mV) for same concentration of MZ and NF using 1M KCl as supporting electrolyte. It was observed that with increase in pH, peak potential shift to more negative potential i.e. right side of polarogram for both MZ and NF. For MZ peak height increases with increase in pH till pH = 6 then it decreases with increase in pH. For NF there was no peak till pH = 4. First peak appears at pH = 5 then there was a continuous decrease in peak height with increase in pH. Figure [1] shows overlaid polarograms of MZ and NF combination at various pH (2-10).

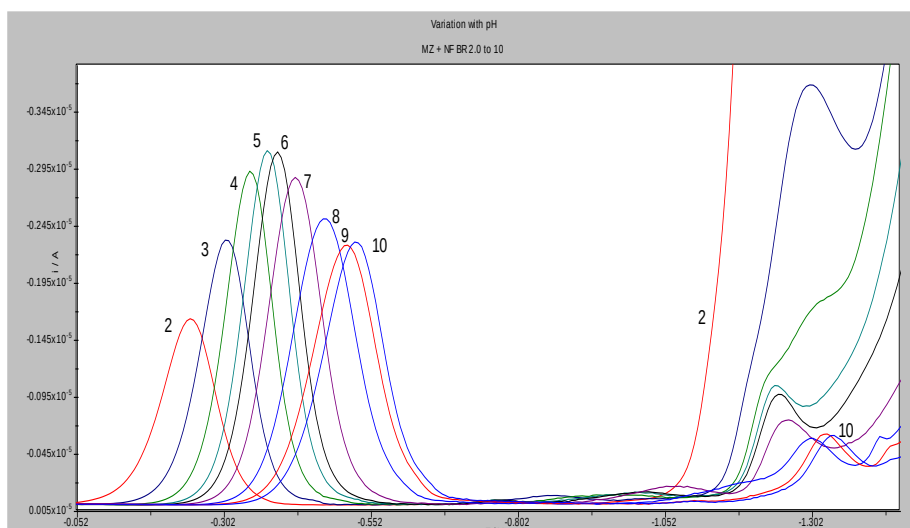


Figure [1] Polarogram of MZ and NF combination at various pH_(2 to 10), with pulse amplitude of 50 mV, and the scan rate of 15mV/s

Optimization of Pulse amplitude:

The peak current varies linearly with the pulse amplitude. polarograms were recorded by changing pulse amplitude from 10mV to 100 mV with the interval of 10mV. The pulse amplitude of 50 mV was chosen for all the analytes, as at higher pulse amplitude the (R^2) values were not satisfactory. While at lower pulse amplitude the response was poor.

Effect of pulse amplitude on polarogram of MZ and NF: -

Polarogram of MZ and NF combination were recorded at different pulse amplitude (10-100 mV) at fixed scan rate (15 mV/s) and at pH = 6.5 for same concentration of MZ and NF using 1M KCl as supporting electrolyte. It was observed that with increase in pulse amplitude, peak potential shifted slightly towards positive side i.e. towards left side of the polarogram for both MZ and NF. Peak height increases continuously with increase in pulse amplitude for both MZ and NF. **Figure [2]** shows overlaid polarograms of MZ and NF combination at various pulse amplitudes (10-100 mV).

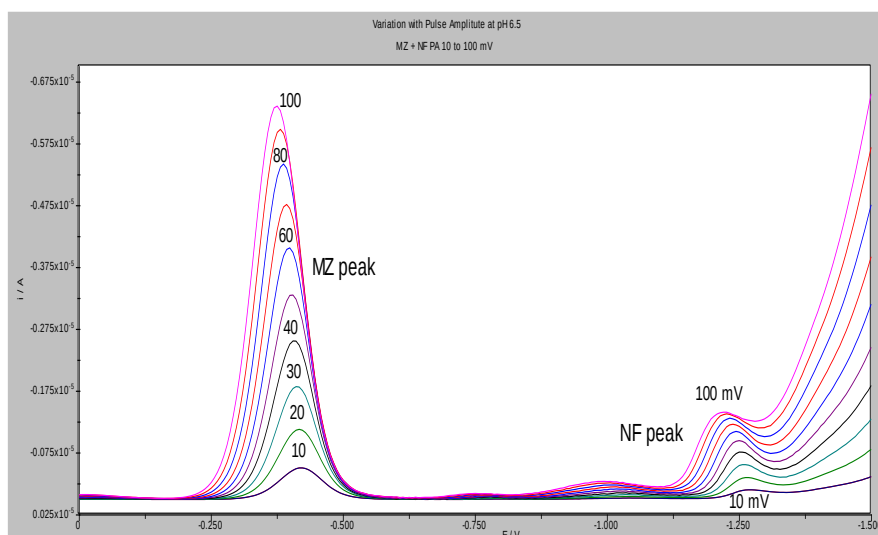


Figure [2] Polarogram of MZ and NF combination at various pulse amplitudes (10 to 100 mV), at pH = 6.5 and at the scan rate of 15mV/s

Optimization of Scan rate:

The polarograms for **MZ** and **NF** combination were recorded at various scan rates from **5 mV/s** to **35 mV/s** with the interval of **5mV/s**. At scan rate higher than **15mV/s** the peak height increased but peak shape of **MZ** got distorted. At a scan rate lower than **15mV/s**, peak height was small, and no proper peak was obtained for **NF**, so a scan rate of **15mV/s** was chosen as the optimum scan rate for the analysis of **MZ** and **NF** combination.

- **Effect of scan rate on polarogram of MZ and NF:-**

Polarogram of **MZ** and **NF** combination were recorded at different scan rate (5-35 mV/s) at fixed pulse amplitude (50 mV) and at pH = 6.5 for same concentration of **MZ** and **NF** using 1M KCl as supporting electrolyte. It was observed that with increase in scan rate, there was no shift in peak potential for both **MZ** and **NF**. Peak height increases continuously with increase in scan rate for both **MZ** and **NF**. At scan rate of 5.0 mV/s no peak was observed for **NF**. **Figure [3]** shows overlaid polarograms of **MZ** and **NF** combination at various scan rates (5-35 mV/s).

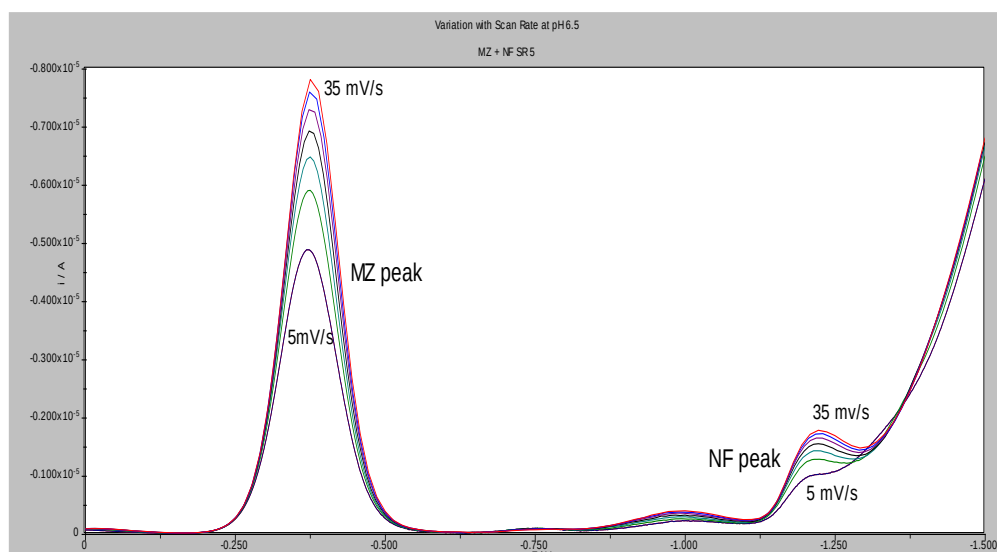


Figure [3] Polarogram of **MZ** and **NF** combination at various scan rates (5 to 35 mV/s), at pH = 6.5 and at the pulse amplitude of 50 mV.

RESULT AND DISCUSSION:

All the optimized voltammetric parameters and instrumental parameters are as follows

Parameters	Optimum Values
Buffer	Britton – Robinson buffer
pH	6.50
Supporting Electrolyte	1 M KCl
Purge Time (Blank)	180 sec
Purge Time (Addition)	100 sec
Equilibration Time	10 sec
Start Potential	0.0 V
End Potential	-1.5 V
Pulse Amplitude	0.05 V
Pulse Time	0.04 sec
Voltage Step	0.006 V
Voltage Step Time	0.4 sec
Scan Rate	0.015 V/sec

CONCLUSION:

The optimized voltammetry parameters such as pH, pulse amplitude and scan rate for Metronidazole and Norfloxacin can be used for any further research

involving electrochemistry of Metronidazole and Norfloxacin.

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