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STUDY OF VARIATION IN ELECTROCHEMICAL BEHAVIOUR OF FURAZOLIDONE AND METRONIDAZOLE SIMULTANEOUSLY BY DIFFERENTIAL PULSE VOLTAMMETRY

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Keywords:

Differential Pulse Voltammetry (DPV), Furazolidone, Metronidazole, Britton-Robinson Buffer, Pulse Amplitude and Scan Rate

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ABSTRACT: In present study, a successful attempt has been made to study the variation in electrochemical behavior of Furazolidone and Metronidazole 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 4.25. Both Furazolidone and Metronidazole exhibited reduction cathodic peak at optimum pH with peak potential at -0.06 V for Furazolidone, and -0.22 V for Metronidazole *vs.* S.C.E. 0.1N CH_3COOH was used as Solvent for the analysis. The variation in electrochemical behavior of Furazolidone and Metronidazole simultaneously at the optimized pH was studied by varying pulse amplitude and scan rate. The optimized pulse amplitude was found to be 50 mV, and the optimized scan rate was found to be 15 mV/s. This study can be used developing methods for simultaneous detection and quantification of Furazolidone and Metronidazole.

INTRODUCTION: The 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 has been reported [5-7]. The simultaneous determination of some combinations by electroanalytical method has been reported [8-12]. 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.

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It can also be used for devising electo-sensors for those pharmaceutical drugs. Furazolidone, $C_8H_7N_3O_5$ that is 3-{[(5-nitro-2-furyl) methylene] amino}-1, 3-oxazolidin-2-one, (Molecular Weight: 225.16 g/mol), it has been used to in the treatment of diarrhoea caused by bacteria or protozoan infections. Metronidazole, $C_6H_9N_3O_3$ that is 2-(2-methyl-5-nitro-1H-imidazol-1-yl) ethanol, is an antibiotic, amebicide, and anti-protozoal (Molecular weight: 171.15 g/mol g/mol) It is highly effective for bacterial and protozoan infections and is available in the tablet form. Furazolidone and Metronidazole in the combined dosage form are available in the market, it is used for treating diarrhea, bacterial and protozoal infections.

OBJECTIVE: The main objective of the study is to provide optimized parameters such as pH, pulse amplitude, and scan rate of volammogram for Furazolidone and Metronidazole simultaneously which can be used in the method development and validation of Furazolidone and Metronidazole in

combined pharmaceutical formulations using Differential Pulse Voltammetry technique.

MATERIALS AND METHODS:

Introduction to 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 Euiptrances model no. 610.

FIG. 1: ELECTROCHEMICAL WORKSTATION- PG STAT 30

FIG. 2: 663 VA ELECTRODE STAND (METROHM)

Solution Preparation:

Combined Stock Solution of Standard FRZ and Standard MZ (50 µg/mL + 150 µg/mL): 25 mg of standard FRZ and 75 mg standard MZ was accurately weighed and transferred into 500 mL standard flask, about 450 mL of 0.1 N 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 500 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 FRZ and MZ combination was studied over the pH range 2 to 10 in Britton – Robinson buffer. Both FRZ and MZ gave a very good linear response with increase in concentration from pH = 3 to pH = 6, from pH = 7 linear response of FRZ peak was not satisfactory. Two peaks of FRZ and MZ are not well resolved at pH = 2 and pH = 3.0. From pH ≥ 4.0 the two peaks are properly resolved. For pH ≥ 4.5 FRZ peaks show positive shift, *i.e.* towards left side with increase in concentration of FRZ. pH = 4.25 was found to be optimum pH due to better-resolved peaks, good R_2 value and there was less shift of FRZ peak with increasing concentration.

Effect of pH on Voltammogram of FRZ and MZ: Voltammogram of FRZ and MZ combination were recorded at different pH (2-10) at fixed scan rate (15.0 mV/s) and at fixed pulse amplitude (50 mV) for the same concentration of FRZ and MZ using 1 M KCl as supporting electrolyte. It was observed that with increase in pH, peak potential shifts to more negative potential, *i.e.* right side of Voltammogram for both FRZ and MZ. The response for both FRZ and MZ was very good for all values of pH. For FRZ, peak height remains constant for almost all pH values except at pH = 2.0 when two peaks are merged. There was marginal increase in peak height of MZ peak with increase in pH till pH = 5; then peak height roughly remains constant with increase in pH. **Fig. 3** shows overlaid Voltammograms of FRZ and MZ combination at various pH (2-10).

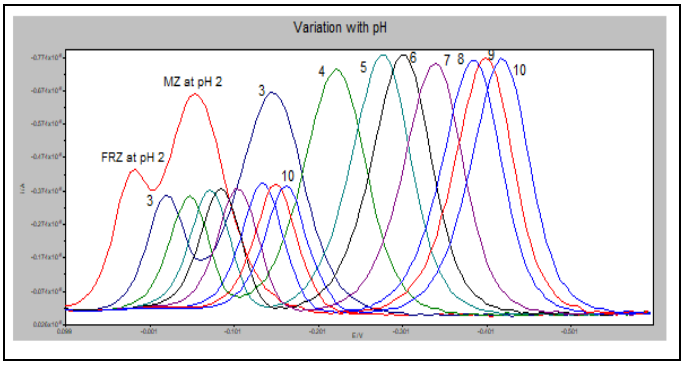

FIG. 3: VOLTAMMOGRAM OF FRZ AND MZ COMBINATION AT VARIOUS pH_(2 TO 10), WITH PULSE AMPLITUDE OF 50 MV, AND THE SCAN RATE OF 15.0 MV/S

Optimization of Pulse Amplitude: The peak current varies linearly with the pulse amplitude in the range of 10 mV to 100 mV. The pulse amplitude of 50 mV was chosen for all the analytes because (R_2) values were not satisfactory at higher pulse amplitudes and response was poor at lower pulse amplitudes.

Effect of Pulse Amplitude on Voltammogram of FRZ and MZ: Voltammogram of FRZ and MZ combination were recorded at different pulse amplitude (10-100 mV) at fixed scan rate (15.0 mV/s) and pH = 4.25, for the same concentration of FRZ and MZ using 1 M 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 Voltammogram for both FRZ and MZ. The shift for FRZ was more prominent than MZ. Peak height increases continuously with increase in pulse amplitude for both FRZ and MZ. **Fig. 4** shows overlaid Voltammograms of FRZ and MZ combination at various pulse amplitudes (10-100 mV).

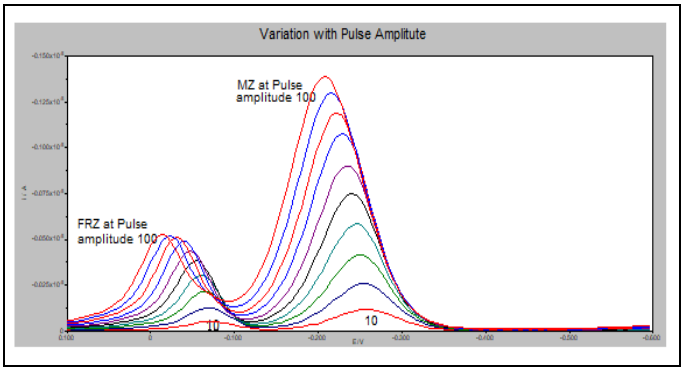

FIG. 4: VOLTAMMOGRAM OF FRZ AND MZ COMBINATION AT VARIOUS PULSE AMPLITUDES (10 TO 100 MV), AT pH = 4.25 AND AT THE SCAN RATE OF 15.0 mv/s

Optimization of Scan Rate: The Voltammograms for FRZ and MZ combination were recorded at various scan rates from 5 mV/s to 35 mV/s with an interval of 5 mV/s. At scan rate of 5 mV/s and 10 mV/s the response was very low with unsatisfactory R2 values. At higher scan rate ≥ 20.0 mV/s distorted peaks were observed. The scan rate of 15.0 mV/s was chosen as the optimum scan rate because it gave better peak shape along with good R2 values.

Effect of Scan Rate on Voltammogram of FRZ and MZ: Voltammogram of FRZ and MZ combination were recorded at different scan rate (5-35 mV/s) at fixed pulse amplitude (50 mV) and at pH = 4.25 for the same concentration of FRZ and MZ using 1M KCl as supporting electrolyte. It was observed that with increase in scan rate, there was no shift in peak potential for both FRZ and MZ. Peak height increases continuously with increase in scan rate for both FRZ and MZ. Distorted Peak shapes were observed at scan rate equal to and greater than 20 mV/s for both FRZ and MZ. **Fig. 5** shows overlaid Voltammograms of FRZ and MZ combination at various scan rates (5-35 mV/s).

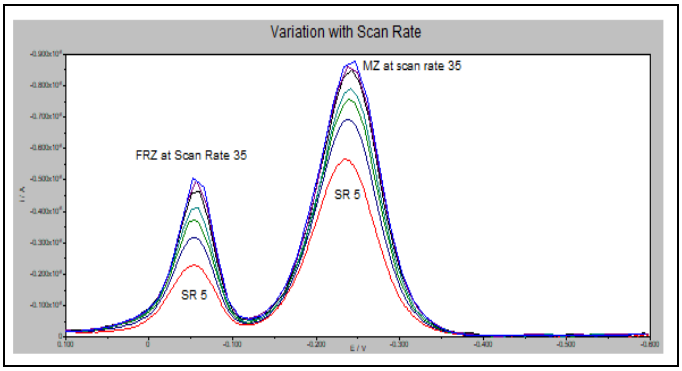

FIG. 5: VOLTAMMOGRAM OF FRZ AND MZ COMBINATION AT VARIOUS SCAN RATES (5 TO 35 MV/S), AT pH = 4.25 AND AT THE PULSE AMPLITUDE OF 50 mv

RESULTS AND DISCUSSION:

All the Optimized Voltammetric Parameters and Instrumental Parameters are as Follows:

Parameters	:	**Optimum values**
Buffer	:	Britton – Robinson buffer
pH	:	4.25
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	:	-1.0 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 voltammetric parameters such as pH, pulse amplitude and scan rate for Furazolidone and Metronidazole can be used for any further research involving electrochemistry of Furazolidone and Metronidazole.

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CONFLICTS OF INTEREST: Nil

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