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Spectrophotometric Determination of Moxifloxacin Hydrochloride using Palladium (Pd-II) ions in micellar medium

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A B S T R A C T

A simple ,sensitive and accurate spectrophotometric method was described for the determination of Moxifloxacin hydrochloride (MFX) a broad spectrum flouro quinolone anti bacterial either in pure form or in the tablet .The method is based on chelate formation between MFX and Palladium(Pd II) in aqueous media at pH 8 in presence of surfactant . The complex showed an absorption maximum at 370nm for zero order, Ist derivative at 437 nm and Second derivative at 457 nm respectively with apparent molar absorpitivity of $1.37x10^4$ $L\text{-}M^{-1}Cm^{-1}$and sandell's sensitivity of 0.00364 $\mu g/cm^2$ respectively. The solution of the complex obeyed beer's law in the concentration range of 1-25 µg/ml for zero order, 10 to50 µg/ml for Ist order and 1to 25 µg/ml for 2nd order respectively. The limit of detection and limit of quantification were calculated and RSD were calculated. The chelate composition between MFX and Pd(II) ion was found to be 1:1 ratio determined by Job's continuous method and by Molar ratio method. The proposed method was applied for the determination of MFX in tablets without interference from common excipients. The results obtained by the application of this procedure showed good percentage recoveries.

Keywords: Flouroquinolone, Moxifloxacin, Hydrochloride, Chelate, Aqueous media, Spectrophotometric, Pharmaceutical formulation

A R T I C L E I N F O

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1. Introduction

Moxifloxacin (MFX) chemically 1-cyclopropyl-7-((s, s)-2,8- diazabicyclo(4.3.0)non-8-yl)-6-Fluoro-8-methoxy-1,4-dihydro-4-oxo-3quinoline carboxylic acid, A new flouroquinolone antibacterial compound Moxifloxacin can be used to treat respiratory infections, including acute sinusitis, acute exacerbations of chronic bronchitis, and community- acquired pneumonia, as well as skin and skin structure infections. Moxifloxacin is used as a second-line agent in tuberculosis (TB) and may potentially have benefits in reducing treatment duration from its current six month to four months. In ophthalmology, Moxifloxacin is available in the form of eye drops, to treat conjunctival infections caused by susceptible bacteria and to prevent infection following eye surgeries. Only few methods for the analysis of MFX are available which include derivative spectroscopy in micellar medium, simple UV spectrophotometric methods, HPLC methods, HPTLC, LC/MS/MS Differential pulse polarography [15], capillary electrophoresis [16].

Complexes of drugs with Palladium (III) have also been studied with Palladium (II) is one of the transitional element which was reported to form stable complexes with many drugs such as Phenothiazines [17,18] captopril [19], N-acetyl L-Cysteine [20] and Timonacic [21].

The purpose of this present study was to develop direct and derivative spectrophotometric, stability indicating procedure for the selective determination of MFX by chelation with Pd(II) ions, to develop procedure capable of quantitation. The methods based on chelation of the drug with Palladium have not been studied and prospective work will be the study using proposed chelation procedures by direct and derivative spectroscopy which is a useful technique that resolves two overlapping spectra and eliminating matrix interference in the mixture of components [22-24].

2. Experimental

Apparatus:

All absorption Spectra were made using Schimadzu-160 A U.V-VIS Spectrophotometer equipped with 10mm matched Quartz cells.

Materials and reagents

Palladium chloride ($Pdcl_2$) $1x10^{-2}$M solution in distilled water was prepared. MFX $1x10^{-3}$M solution was prepared by dissolving 43.8mg of MFX into a 100ml volumetric flask. It was dissolved and diluted upto the mark using double distilled water and sodium dodecylsulphate (1.2 ml of 0.1M) to get $1x10^{-3}$M concentration of MFX solution.

Chelation of Mfx with Pd (II):

The following procedure was adopted for measuring the absorption spectra of complex (metal + drug) in aqueous medium. In a 10-ml standard flask, 1ml of $1x10^{-3}$M MFX stock solution the metal complex was prepared by taking 3 ml of buffer, suitable volume of surfactant, suitably concentration of Palladium ion metal solution (usually 10-15 fold molar excess to drug) solutions. The contents were diluted up to the mark with distilled water and the absorbance of the complex was measured against the reagent blank prepared identically. A plot between absorbance and the wavelength was plotted from which the analytical wavelength was selected. The ƛ-max for the Chelate at Zero order is 370 nm, first order derivative is obtained at 437 nm and second order derivative at 457 nm. Figs 1-3.

Procedure for dosage form

An accurately weighed amount of finely powdered tablet equivalent to 100mg of drug was dissolved in about 10ml of distilled water and transferred in to 100ml of calibrated volumetric flask and 1.2 ml of 0.1 M sodium dodecyl sulphate was added, after 15 minutes mechanical shaking was filtered into a 100ml of calibrated volumetric flask through Whatmann no:41filter paper ,diluted to 100ml with distilled water and the same procedure was followed as described above.

Optimum conditions

Effect of pH

To arrive the optimum pH required for achieving the maximum and constant absorbance, the effect of pH on the absorbance of the Palladium (II)–MFX complex was studied by employing in a set of 10-ml standard flasks, 3 ml of buffer (different pH values 1.0 to 11.0) solution, constant amount of drug and metal ion (usually 10-15 fold molar excess to drug) solution were taken, made up to the mark with distilled water. The absorbance of each solution (metal complex) was measured at a selected wavelength (λ_{max}) against corresponding reagent blank prepared accordingly. A plot was made between absorbance and pH from which the working pH was selected. The complex shows maximum and constant absorbance in the pH range 8.0. Therefore, buffer solution having pH 8.0 was chosen for further studies. Fig 4.

Effect of reagent concentration

To 1ml of $1x10^{-3}$ M MFX stock solution, aliquots of 0.5to 3ml of $1x10^{-2}$M reagent solution (Pd II) was added into 10 ml Volumetric flask and make upto the volume to 10ml with distilled water and the absorbance values at 370nm. Investigation of metal ion concentration revealed that only eight-fold excess of reagent was sufficient for optimum and maximum colour intensity of the chelate of MFX using 43.8µg/ml concentration Fig 5.

Effect of time

The absorbance of MFX-Pd(II) complex was measured at different time intervals to ascertain the time stability of the complex. The full colour development of the complex remains constant for twenty four hr. Then the absorbance of MFX-Pd(II) complex was measured at 370 nm.

Determination of chelate stability and composition

The composition of the chelate of MFX with Pd(II) ion used was studied by Job's continuous method and Molar ratio method .The chelate of 1:1 ratio was obtained between MFX and Pd(II). Fig 6& Fig 7. The stability constants of formed chelate were calculated and the values of Log β was 6.88.The results were tabulated in Table 1. [31-36]

Linearity range and quantification procedure

Beer's law was found to be obeyed in the concentration range of 1to 25µg/ml for Zero order,10 to 50µg/ml for Ist order derivative and 1to 25µg/ml for 2^{nd} order derivative

.A(1%,1Cm) was calculated .The results were tabulated in Table 2.

Assay of dosage form [37-46]

An accurately weighed amount of finely powdered tablet equivalent to 100mg of drug was dissolved in about 10ml of distilled water and transferred in to 100ml of calibrated volumetric flask, 1.2 ml of 0.1 M sodium dodecyl sulfate was added and after 15minutes of mechanical shaking was filtered into a 100ml of calibrated volumetric flask through Whatmann no: 41 filter paper and was diluted to 100ml with distilled water and the same procedure was followed as described above. The results were tabulated in Table 3.

Interference study

Potential interference by the excipients in the dosage form was also studied, samples were prepared by mixing fixed amounts of common excipients such as lactose, Micro crystalline cellulose, Talc, Magnesium sterate and Starch. The good percentage recoveries were obtained indicating no interference was observed. The results were tabulated in Table 4.

3. Results and Discussion

The linearity range of MFX-Pd(II) chelate covered over a range of 1-25 µg /ml of drug with A(1%,1cm)equals to $1.37x10^4$ L $Mole^{-1}cm^{-1}$.The drug chelate absorbance were plotted against the corresponding concentrations. Data were fitted to the equation Y=a+bx, where Y is the absorbance at relevant maximum is the Drug concentration in mcg/ml; b is the slope and a is the intercept of the calibration curve. The correlation coefficient is 0.999 indicating exact linearities.

The Accuracy of the proposed procedure was 100%. Repeatability and reproducibility were evaluated. Proposed procedure for MFX is a stability indicating one which can be used for the determination without interference with the excipients. The drug being soluble in presence of surfactant in aqueous medium and considered more selective drug to chelate with Pd(II) ion, in addition, the derivative spectra normally contain more apparent spectral details than the normal spectra, more selective and sensitive in eliminating the background interference of complex matrix in resolving individual drug, drug additives and drug decomposition both interfered.

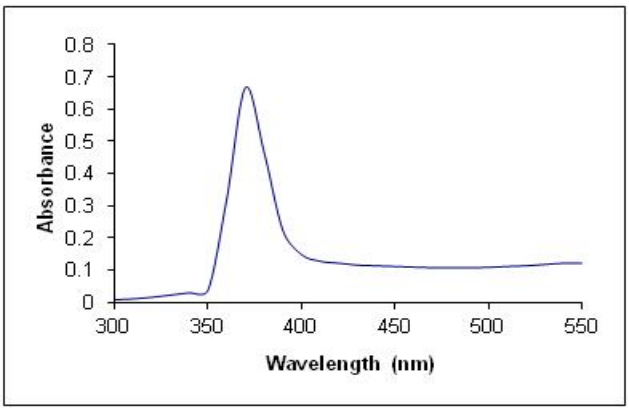

Figure1: Absorption spectra of 43.8µg/ml MFX complex with $1x10^{-2}$M Pd(II).

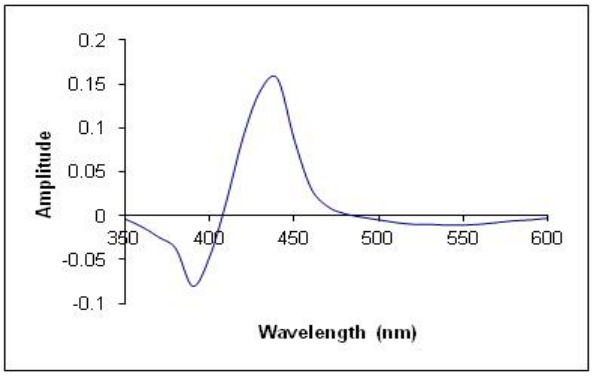

Figure 2: First order derivative spectra of43.8µg/ml MFX complex with $1x10^{-2}$M Pd (II).

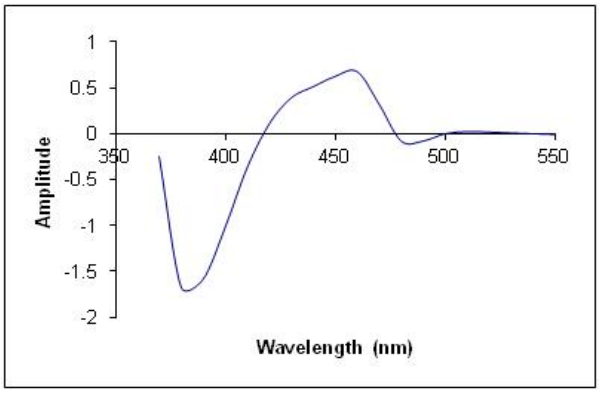

Figure 3: Second order derivative spectra of of43.8µg/ml MFX complex with $1x10^{-2}$M Pd (II).

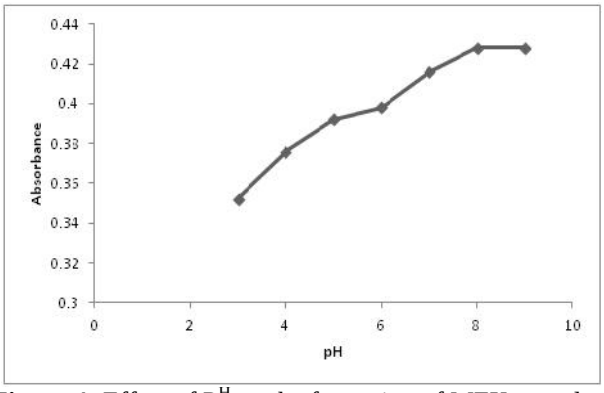

Figure 4: Effect of P^H on the formation of MFX complex with Pd (II) ion.

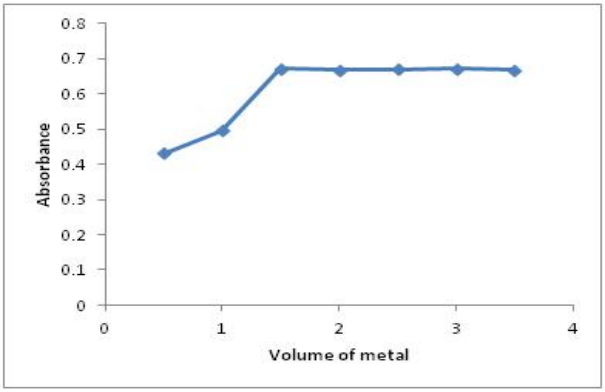

Figure 5: Effect of reagent concentration on the formation of MFX complex with Pd(II)ion.

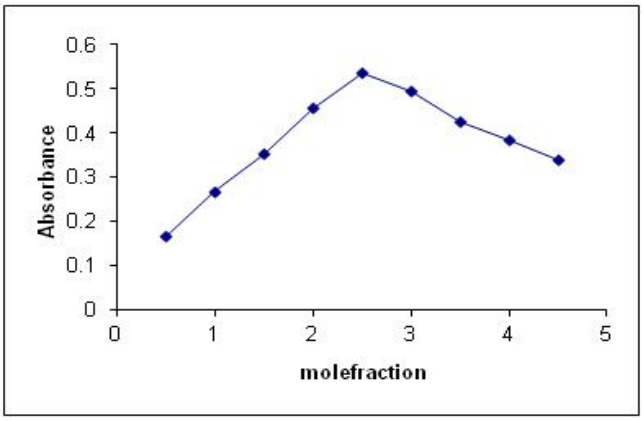

Figure 6: Job's method for MFX complex with Pd (II) ion at 370nm.

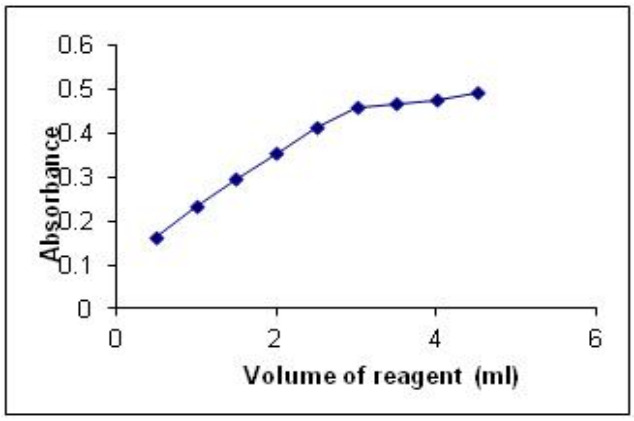

Figure 7: Mole ratio method for MFX complex with Pd (II) ion at 370nm.

Table 1 Stability constants of Moxifloxacin hydrochloride chelate with Pd(II) by Job's method.

Parameters	**MFX-Pd(II) λmax at 370nm**
Total molar conc	$1x10^{-5}$M
N	2.303
β*	$7.572x10^{6}$
Logβ	6.88

Table 2 Results of validation

Parameter	**MFX-Pd(II) 368nm**	**MFX-Pd(II) Ist derivative**	**MFX-Pd(II) 2nd derivative**
Linearity range(µg/ml	1-25	10-50	1-25
LOD (mcg/ml)	0.19	0.421	0.49
LOQ (mcg/ml)	0.58	1.25	1.3
Slope	0.040	0.021	0.026
Intercept	0.029	0.011	0.029
Correlation coefficient	0.998	0.999	0.999
Accuracy	100.01	100.13	100.3
Repeatability(n=6)	0.1067	0.1792	0.1063

Table 3: Results of the determination of MFX by the proposed method in their dosage form.

Dosage Form	**MFX-Pd(II) At 370nm**	**MFX-Pd(II) At 437**	**MFX-Pd(II) At 457**	**LIMIT**
Tablet 1	100.03±0.1105 N=6	100.1±0.1730 N=6	100.2±0.1569 N=6	The Assay of Moxifloxacin Hydrochloride tablets should be within 98%-102%
Tablet 2	100.00±0.1414 N=6	100.08±0.1770 N=6	100.2±0.1767 N=6	

Table 4: Determination of Moxifloxacin in presence of common excipients by the proposed method.

	Recovery ±RSD[a]	**Recovery±RSD[a]**	**Recovery±RSD[a]**
Excipient	**At 370nm**	**1st derivative At437nm**	**2nd derivative at 457nm**
Lactose(10mg)	99.96±0.075	100.1±0.1342	100.2±0.1629
Talc(10mg)	100.01±0.1067	100.1±0.1153	100.2±0.1767
Magnesium sterate(10mg)	100.0±0.1290	100.1±0.1412	100.17±0.1595
Starch(10mg)	99.98±0.1343	100.1±0.1211	100.23±0.1967
Microcrystalline cellulose	100.01±0.1343	100.13±0.1372	100.2±0.2110

a-Values are mean of six determinations.

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