International Journal of PharmTech Research
CODEN (USA): IJPRIF ISSN : 0974-4304
Vol.5, No.1, pp 198-204, Jan-Mar 2013

Quantitative Estimation Of Cefixime And Moxifloxacin In Pharmaceutical Preparation By UV Spectrophotometric Method

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Abstract: A simple, accurate, precise, reproducible UV Spectrophotometric methods have been developed for the simultaneous estimation of Cefixime and Moxifloxacin in bulk and its synthetic Mixture. First order derivative spectrophotometry method, wavelengths selected for quantitation was 259 nm for Cefixime (zero crossing point for Moxifloxacin) and 380 nm for Moxifloxacin (zero crossing point for Cefixime). In this method linearity was observed in the concentration range of 2-10 µg.mL-1 for Cefixime as well as Moxifloxacin. The results of analysis were validated statistically and by recovery studies. The proposed method was found to be successful for the simultaneous estimation of both drugs in bulk and its synthetic mixture. The results of analysis have been validated statistically for linearity, accuracy, precision, LOD and LOQ of proposed method.

Key Words: Cefixime, Moxifloxacin, First order derivative, UV spectrophotometry.

INTRODUCTION

Cefixime (CEFI) is official in British pharmacopoeia. It is chemically 8-[[2-(2-amino-1, 3-thiazol4-yl)-2-(carboxymethoxyimino)acetyl] amino]-4-ethnyl-7-oxo-2-thia-6-azabicyclo [4.2.0] oct-4-ene-5-carboxylic acid (Figure 1). It is used in the treatment of otitis media, respiratory tract infections and urinary tract infections caused by susceptible organisms. Moxifloxacin (MOXI) is official in British pharmacopoeia. It is chemically 1-Cyclopropyl-6-fluoro-8-methoxy-7-[(4a*S*, 7aS)-octahydro-6*H*pyrrolo [3,4*B*] pyridin-6-yl]-4-oxo-1, 4-dihydroquinoline-3-carboxylic acid hydrochloride (Figure 2). Use in Ocular infection, acute sinusitis, Lower respiratory tract infections, UTI. A formulation containing 400 mg of CEFI and 400 mg of MOXI (SR) tablet Approved by CDSCO in August 2011. Many Works has been done on individual plain tablets. A survey of literature revealed that few chromatographic and Spectrophotometric, HPLC and HPTLC; Densitometric methods are reported for determination of CEFI and MOXI individually [8-27]. However there is no method reported so far determination of CEFI and MOXI in its combine dosage form. The present work describes a validated, simple, precise and accurate spectrophotometric method for estimation of CEFI and MOXI in its combined dosage formulation.

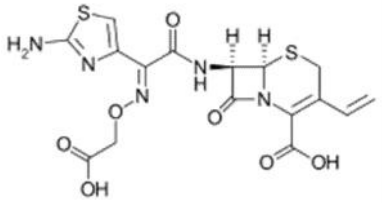

Figure 1: Structure of CEFI

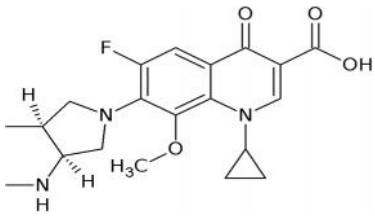

Figure 2: Structure of MOX

MATERIALS AND METHOD

Apparatus

The instrument used for the present study was a UV-Vis double beam spectrophotometer (model 2080, Analytical Technological Limited) with 1cm matched pair quartz cell, Electronic weighing balance. Calibrated glass wares were used throughout the work.

Reagents and Materials

The drug, CEFI is procured as a gratis sample from Cipla, Goa branch and MOXI from Matrix, Hyderabad branch. The solvent system used for the entire analysis was Methanol: Water (20:80), both were of AR grade, purchased from SD Fine Chemicals Limited, India and double distilled water.

Preparation of standard stock solution

An accurately weighed quantity of CEFI (100mg) and MOXI (100mg) were transferred to a separate 100ml volumetric flask and dissolved and diluted up to the mark with solvent system (1000 µg.mL-1). From this 10ml pipetted out and transferred in to another 100ml volumetric flask, then diluted up to mark with solvent to obtained stock solution (100 µg.mL-1).

METHOD

Stock solutions of both drugs were diluted suitably scanned separately in the range of 200-400 nm against solvent blank. The zero order spectra (Figure 3) showing a complete overlapping necessitates advanced method. Hence it is derivatised from first to fourth order. Out of these first order is considered for analysis. From the overlain spectra of both drugs and mixture (Figure 4) wavelength 259 nm (zero crossing point MOXI) for CEFI and 380 nm (zero crossing point CEFI) for MOXI were selected for quantification of both the drugs. The standard stock solution was diluted suitably to prepare concentration range of 2-10 µg.mL-1 for CEFI and MOXI in solvent using working standard solution (100µg.mL-1), then scanned and derivetised. At 259 nm calibration curve was plotted for CEFI which shows no interference from MOXI. And at 380nm calibration curves plotted for MOXI which shows no interference from CEFI. Statistical data for the calibration curve is as depicted in Table 1. Linearity is obtained individually and combination within the concentration range of 2-10µg.mL-1 for CEFI and MOXI both.

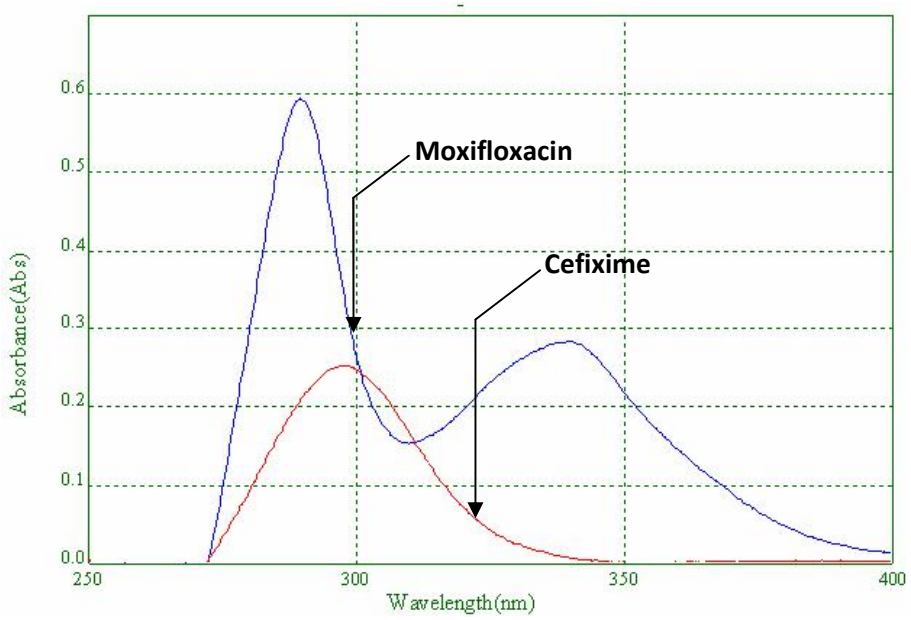

Figure 3: 1st Order Overlain spectra of CEFI, MOXI

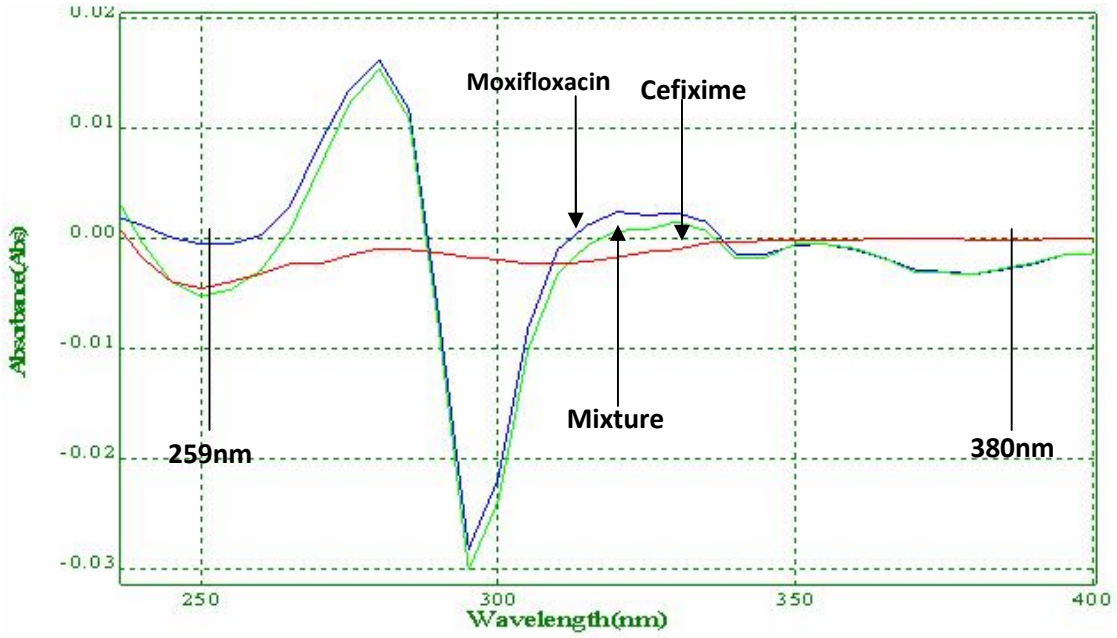

Figure 4: 1st Order Overlain spectra of CEFI, MOXI, and Mixture

Table 1: Linear regression analysis of calibration curves*

	CEFI	MOXI
Wavelength (nm)	259.0	380.0
Beer's law limit (µg/ml)	2-10	2-10
Slope	0.00077	0.00076
Intercept	0.0012	0.00024
Correlation Coefficient	0.9993	0.9993
LOD (µg.mL-1)	0.092	0.088
LOQ (µg.mL-1)	0.280	0.268

*Average of six determinations

Application of the Proposed Methods for the determination of CEFI and MOXI in synthetic mixture

In order to further assess the applicability of the proposed method for the determination of the selected drugs in their binary solutions, synthetic mixtures were prepared mixing the aliquot portions of individual stock solutions to get final concentration of CEFI (10 µg.mL-1) and MOXI (10 µg.mL-1) respectively. The acceptable recovered concentrations, values of SD (%) compiled in Tables 3 and 4 for CEFI and MOXI mixtures respectively, confirm the accuracy and precision of the method, and demonstrate its analytical power to resolve and quantify the investigated drugs.

VALIDATION OF ANALYTICAL METHODS [5-6]

The developed method was validated statistically as per ICH guidelines for all the parameters. Like accuracy, linearity, precision, ruggedness and specificity. Accuracy of the method was established on the basis of recovery studies, carried out by standard addition method in which pre-analyzed samples were taken and standard drug was added at three different levels (80%, 100% and 120% of the test concentration). Result of recovery studies and percentage recovery were found to be satisfactory and are reported in **Table 2.** The linearity of the method was established from the first derivative spectra by measurement of absorbance of standard solutions containing varying concentrations of each compound. Linearity was found for the range of 2-10 $\mu g.mL^{-1}$ (r^2 <1). Precision of the method was demonstrated by intraday and interday variation studies. In intraday variation study three replicates of solutions at three concentration level 2, 6, 10 $\mu g.mL^{-1}$ were carried out. Similarly intraday precision was also carried out by repeating analysis for three days and concentrations were calculated

results were ascertained by % RSD < 2. Ruggedness was established by carrying out experiment at different conditions by different analyst. Specificity of the method was ascertained by analyzing standard drug in presence of excepients, which shows no interference at all. **(Table-3)**

Application of the Proposed Methods for the determination of CEFI and MOXI in synthetic mixture

In order to further assess the applicability of the proposed method for the determination of the selected drugs in their binary solutions, synthetic mixtures were prepared mixing the aliquot portions of individual stock solutions to get final concentration of CEFI (10 µg.mL-1) and MOXI (10 µg.mL-1) respectively. The concentration of both CEFI and MOXI were determined by measuring the absorbance of sample at 259.0 nm and 380.0 nm in first order spectrum mode respectively and the results of synthetic mixture analysis were calculated from the calibration curve in quantitation mode.

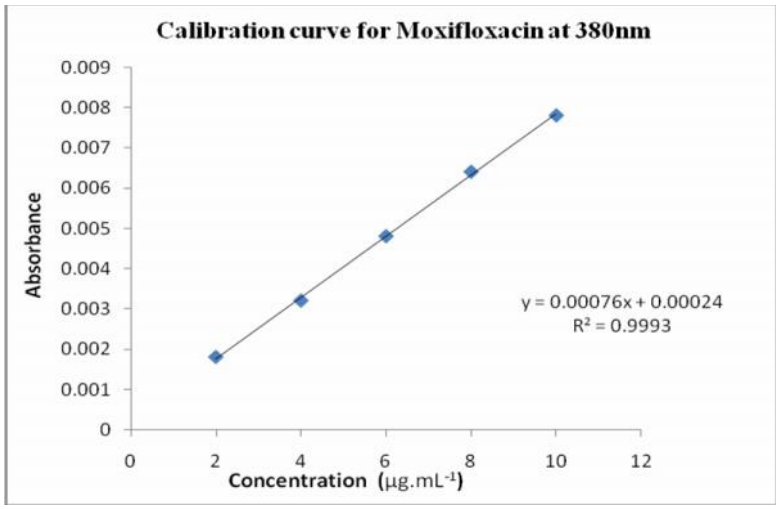

Figure 5: Calibration Curve of MOXI at 380nm

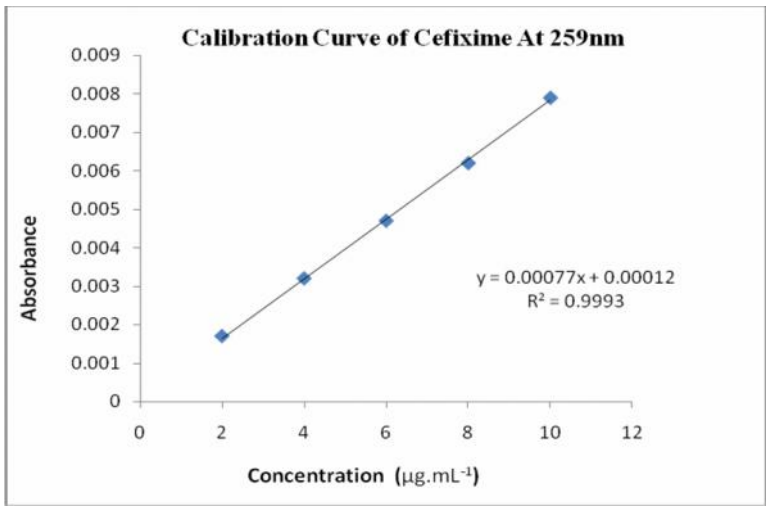

Figure 6: Calibration Curve of CEFI at 259nm

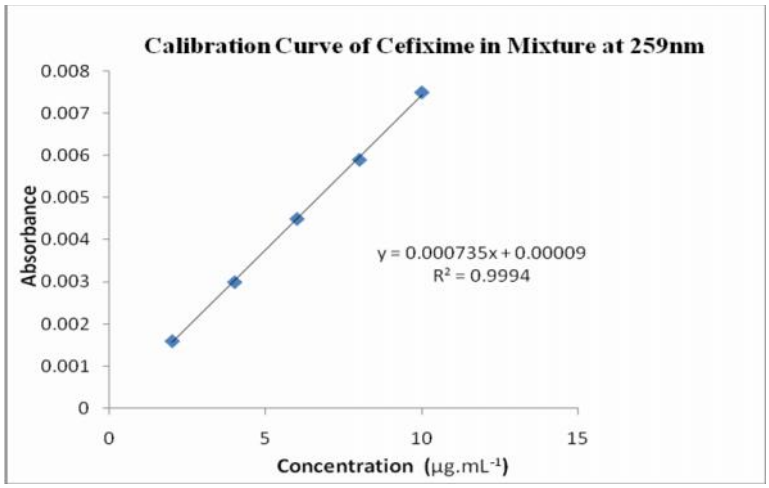

Figure 7: Calibration Curve of CEFI in Mixture at 259nm

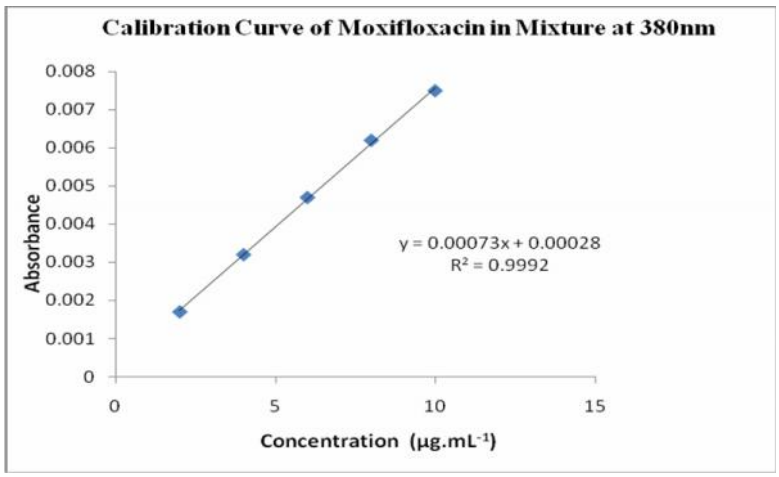

Figure 8: Calibration Curve of MOXI in Mixture at 380 nm

Table 2: Recovery studies of CEFI and MOXI in Mixture

Drug	Amount Added (µg/ml)	Total Amount Found(µg/ml)	% Recovery	Average % Recovery	%RSD
CEFI (2 µg.mL-1)	1.6	3.6	98.63	100.28	1.72
	1.6	3.6	102.41		
	1.6	3.6	100.15		
	2	4	98.97		
	2	4	99.31		
	2	4	103.74		
	2.4	4.4	99.87		
	2.4	4.4	100.49		
	2.4	4.4	98.94		
MOXI (2 µg.mL-1)	1.6	3.6	99.31	100.69	1.29
	1.6	3.6	100.17		
	1.6	3.6	98.45		
	2	4	102.73		
	2	4	100		
	2	4	101.36		
	2.4	4.4	102.16		
	2.4	4.4	100.45		
	2.4	4.4	101.59		

Table 3: Summary of Validation Parameter of proposed method

Parameters	CEFI	MOXI
Linearity (µg.mL-1)	2-10	2-10
Intraday precision (% RSD)	1.53	0.88
Interday precision (% RSD)	1.52	1.16
Ruggedness	1.58 ± 2%	0.85 ± 2%
Robustness	1.67 ± 2%	1.29 ± 2%
Specificity	100 ± 2%	100 ± 2%

Table 4: Result of analysis of synthetic mixture

METHOD	MIXTURE	Labeled claim (mg)	Amount found* (mg)	% Label claim*	± S.D*
1ST DVT	CEFI	400	409.04	100 - 102.26	± 1.30
	MOXI	400	399.36	98 - 100.91	± 1.15

*Mean of three determinations

RESULTS AND DISCUSSION

Linearity was determined at different concentration, CEFI and MOXI shows linearity in the concentration range of 2-10 µg.mL-1 for CEFI and MOXI both. The percent recovery both the drugs are within the rage 95-105% which indicates the method is accurate. The % RSD values for precision are < 2.0%. Method shows a good specificity, ruggedness and robustness. The results of the synthetic mixture were found to be 98.63-103.74% and 98.45-102.73% for CEFI and MOXI respectively. The proposed first derivative method found to be simple, rapid and sensitive. Therefore, validated UV spectrophotometric method will play a role for determination of CEFI and MOXI in their combined dosage formulation.

ACKNOWLEDGEMENT

The authors are grateful to CIPLA Goa Branch, India, and MATRIX, Hyderabad, India for providing gift samples of CEFIXIME and MOXIFLOXACIN respectively.

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