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Research Article

DEVELOPMENT AND VALIDATION OF SIMULTANEOUS EQUATION METHOD FOR THE ESTIMATION OF CEFIXIME AND OFLOXACIN IN PURE AND IN TABLET FORMULATION

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ABSTRACT
This paper describes developed and validated UV spectrophotometry method for the simultaneous estimation of Ofloxacin and Cefixime in a combined dosage form by Simultaneous equation method. This method utilize methanol as solvent. In this method UV spectra of Ofloxacin and Cefixime were recorded, from the overlay spectra the wavelength selected for the simultaneous equation method were 235nm for Ofloxacin and 290nm for Cefexime, where the linearity ranges for both the drugs were 5-30 µg / ml. The correlation coefficient of Ofloxacin and Cefexime was found to be 0.9999 and 0.9998 respectively. The results of analysis of this method have been validated statistically and recovery studies confirmed the accuracy of the proposed method.
KEYWORDS: Ofloxacin , Cefexime , and UV

INTRODUCTION

Ofloxacin is a broad - spectrum antibiotic that is active against both Gram-positive and Gram-negative bacteria. Chemically it is (+)-9-fluoro-2,3-dihydro-3-methyl-10-(4-methyl-1-piperazinyl)-7-oxo-7*H*-pyrido[1,2,3-*de*]-1,4-benzoxazine-6-carboxylic acid. It functions by inhibiting DNA gyrase, a type II topoisomerase, and topoisomerase IV, which is an enzyme necessary to separate replicated DNA, there by inhibiting cell division. The fluroquinolones interfere with DNA replication by inhibiting an enzyme complex called DNA gyrase. Cefixime is generally classified as a third-generation cephalosporin antibacterial, and chemically it is 7-[2-(2-Aminothiazol-4-yl)-2(carboxymethoxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid trihydrate and is given by mouth in the treatment of susceptible infections including gonorrhea, otitis media, pharyngitis, lower respiratory-tract infections[1]. The extensive literature survey carried out revealed that there is no method reported for the simultaneous estimation of these drugs, some methods for estimation of individual drugs or with other drugs UV-spectrophotometry [2-6] , RP-HPLC[7-17] are available. Hence present study aim to developing a specific, precise, linear, simple, rapid, validated and cost effective UV- spectrophotometry method for the simultaneous estimation of these drugs in combined dosage forms.

MATERIALS AND METHODS

Experimental

Apparatus

Absorbance was measured and spectra were recorded over the wavelength range of 200-400 nm in two matched quartz cells with a 1 cm path length using a Shimadzu – 1700 Double beam UV – Visible Spectrophotometer. (Shimadzu, Japan).

Reagents and Materials

Ofloxacin and cefixime were generoumacy, gifted by M/S Sucrane Pharmaceuticals PVT Ltd., Theda, HP and the formulation (CEFI-O) containing 200 mg Ofloxacin and 200 mg Cefixime of from local Pharmacy. All the chemicals and reagents used were of AR grade and purchased from Qualigens Fine Chemicals, Mumbai, India.

Preparation of Standard Solution

10mg of Ofloxacin and 10 mg of Cefixime was accurately weighed. A standard stock solution of Ofloxacin and Cefixime (100µg/ml) was prepared in methanol. These solutions were further diluted to obtain a series of concentration ranging from 5 - 30 µg/ml of Ofloxacin and Cefixime.

Selection of wavelength

An ideal wavelength is the one that gives maximum absorbance's and good responses for the drug detected at lower concentration also. UV spectra of Ofloxacin, and Cefixime were recorded and overlapped. From the overlay spectra the wavelength selected for the simultaneous equation method were235nm for Ofloxacin and 290nm for Cefixime. The entire drug shows significant absorbance at wavelength 235nm and 290 nm, Hence both nm was selected for the simultaneous equation method Fig-1.

Linearity and Range

Aliquots of 5– 30 µg/ml of standard solution was prepared and calibrated. The linear regression data showed good linear relationship over a concentration range of 5 to 30 µg /ml for Ofloxacin and Cefixime .

Accuracy

Accuracy of the method was determined by recovery experiments. To a pre analyzed tablet solution, a definite concentration was added and then its recovery was studied. The preanalyzed formulation solution equivalent to 10µg/ml Ofloxacin and Cefixime a known concentration of Ofloxacin and Cefixime containing 80%, 100%, and 120% was added. The absorbance of the resulting solutions was measured at their corresponding wavelengths and the percentage recovery was then calculated.

Ruggedness

Ruggedness is the degree of reproducibility of the results obtained under a variety of conditions, expressed as %RSD. These conditions include different laboratories, analysts, instruments, reagents, days, etc.

Estimation of Formulation

Twenty tablets each containing quantity equivalent to 200 mg of Ofloxacin and 200 mg of Cefixime was weighed, powdered and average weight was calculated. Quantity equivalent to 10 mg of Ofloxacin and Cefixime was weighed and transferred to a 100 ml volumetric flask. The drug was dissolved in methanol with shaking and finally volume was made up to the mark. The solution was filtered through whatman filter paper. The solution was further diluted with methanol. The formulation was assayed with 100µg/ml of the solution of Ofloxacin and Cefixime. The concentration of the drugs was calculated from absorbance obtained using standard calibration graph and percentage purity was calculated.

RESULTS AND DISCUSSION

Simple precise and accurate method was developed for the estimation of Ofloxacin and Cefixime. Estimation of Ofloxacin and Cefixime was achieved by simultaneously using UV-VIS - 1700 pharma spec. The normal spectrum of Ofloxacin and Cefixime was recorded in methanol.From the overlay spectra the wavelength selected for the estimation is 235nm for Ofloxacin and 290nm for Cefixim. Stock solution of Ofloxacin and Cefixime was prepared and dilution was done in the range of 5-30µg/ml. And the linearity was checked in different concentration. The calibration curve was obtained for Ofloxacin and Cefixime in the range of 5-30µg/ml. The correlation coefficient of Ofloxacin and Cefixime was found be 0.9999 and 0.9998 respectively. **(**Table. 1).

Twenty tablets was weighed, powdered and average weight was calculated. Quantity equivalent to 10 mg of Ofloxacin and Cefixim was weighed and the drug was dissolved in methanol. The solution was further diluted with methanol. The formulation was assayed 100µg/ml of the solution. The concentration of the drugs was calculated from absorbance obtained using standard calibration graph and percentage purity was calculated. (Table. 2).

The recovery studies were also carried out to ensure the reproducibility and reliability of the method of adding known amount of standard solution and analysis was carried out as per the formulation procedure. The report of formulation analysis recovery studies are given in (Table.3). Hence it is concluded that the UV methods can be effectively used for the simultaneous estimation of Ofloxacin and Cefixime from pharmaceutical dosage forms.

CONCLUSION

The evaluation of obtained values suggests that the proposed UV Spectrophotometry methods provide simple, precise, rapid and robust quantitative analytical method for determination of Ofloxacin and Cefixime in tablet dosage form. After validating proposed method as per ICH guidelines and Correlating the obtained values with the standard values, satisfactory results were obtained. The sample recoveries in all Formulations were in good agreement with their respective Label claims and they suggested noninterference of formulation excipients in the estimation. Hence, the method can be easily and conveniently adopted for routine estimation of Ofloxacin and Cefixime in tablet dosage form.

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Table 1: Validation Parameters

S.No.	Parameters	Oflaxacin at 235 nm	Cefixime at 290 nm
1.	Calibration range	5-30µg/ml	5-30µg/ml
2.	Regression Coefficient (r^2)	0.9999	0.9998
3.	Slope(m)	0.00655	0.00551
4.	Intercept(c)	-0.00035	-0.00028
5.	Repeatability of absorbance (%RSD)	0.152	0.179
6.	Precision a) Intra day	0.021	1.3
	b) inter day	0.063	1.15

Table 2: Quantification in Combination Formulation

Formulation	Label claim mg/tab	Ofloxacin		Cefixime	
		Amount found mg/tab ± SD*	% Assay ± RSD	Amount found mg/tab ± SD*	% Assay ± RSD*
Ofloxacin 200mg and Cefixime 200mg	200mg each	199.4 ± 0.03	99.76 ± 0.3	199.2 ± 0.07	99.69 ± 0.3

*RSD for Three Determinations

Table 3: Recovery studies

Formulation	Ofloxacin			Cefixime	
	% Drug Added	% Drug Recovered*	% Recovery ± %RSD*	%Drug Recovered*	% Recovery ± %RSD*
Ofloxacin 200mg and Cefixime 200mg	80	80.08 ± 0.34	100.02 ± 0.19	79.87 ± 0.05	99.83 ± 0.06
	100	100.73 ± 0.94	100.73± 0.93	100.15 ± 0.04	100.51 ± 0.33
	120	119.87 ± 0.83	99.89 ± 0.69	120.46 ± 0.27	100.38 ± 0.22

*RSD for Three Determinations

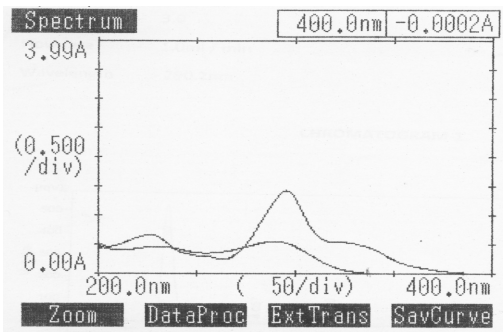

Fig 1: Overlay spectra of Cefixime and Ofloxacin