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Research Article

DEVELOPMENT OF COLORIMETRIC METHOD FOR THE ANALYSIS OF PHARMACEUTICAL FORMULATION CONTAINING BOTH OFLOXACIN AND CEFIXIME

RAJNISH KUMAR *, PINDERJIT SINGH, HARINDER SINGH

State Food, Drug and Excise Laboratory, Punjab, Sector – 11 D, Chandigarh, India, Department of Health and Family Welfare, Punjab
Email: rajnishthaper@gmail.com

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ABSTRACT

An accurate and precise colorimetric method is presented for the determination of ofloxacin and cefixime in same pharmaceutical formulation. Ofloxacin forms an orange colored product in the presence of ferric chloride solution in acidic medium and the absorbance of orange colored species formed was measured at 435 nm against reagent blank and Beer's law was obeyed in the concentration range of 15-75 µg/mL. While cefixime forms a greenish colored product with Fehling solution and the absorbance of greenish colored species formed was measured at 490 nm against reagent blank and Beer's law was obeyed in the concentration range of 5-40 µg/mL. The amount of cefixime and ofloxacin present in the sample was computed from calibration curve. It is also found that there is no interference of cefixime while estimation of ofloxacin and vice versa.

Keywords: Ofloxacin, Cefixime, colorimetric

INTRODUCTION

Ofloxacin chemically is a fluorinated carboxy quinolone, is a racemate, (±)- 9-fluro-2, 3-dihydro-3-methyl-10- (4-methyl-1-piperazinyl)-7-oxo-7H-pyrido [1,2,3-de]-1,4-benzoxazine- 6-carboxylic acid. It is official in BP, USP and EP[1]. Ofloxacin having much greater antibacterial activity than the other urinary tract antiseptics such as nalidixic acid. It inhibits gram negative rods including Enterobacteriaceae, Pseudomonas, Neisseria and other in serum concentratin of 1-5 µg/mL. Gram positive organisms are inhibited at higher concentrations. Ofloxacin act by inhibiting DNA gyrase of microorganisms. Cefixime chemically (6R,7R)-7-[[2-(2-amino-1,3-thiazol-4-yl)-2-(carboxymethyloxyimino)acetyl]amino]-3-ethenyl-8-oxo-5-thia-1- azabicyclo[4.2.0]oct-2-ene-2-carbo.0xylic acid is orally active third generation cephalosporin and used effectively on gram negative range of organisms. It is consistently effective against Enterobacter & Citrabacter and also on beta lactamase producing Haemophilus and Nessieria. The activity of Cefixime against anaerobic bacteria is less impressive[2,3,4]. Cefixime with ofloxacin is used for the treatment of uncomplicated typhoid fever in children[5]. Many HPLC methods are developed for the determination of ofloxacin and cefixime[6,7,8,9]. But HPLC methods are much expensive as compared to the spectrophotometric methods. There are some colorimetric methods developed for the estimation of ofloxacin[10,11] but there is no work in the literature reported about the colorimetric method for the determination of ofloxacin and cefixime in same pharmaceutical formulations. Hence, the authors have made an attempt to develop a simple and rapid colorimetric method for the analysis of pharmaceutical formulations containing both cefixime and ofloxacin.

MATERIAL AND METHODS

Apparatus

A Systronics model 106 digital spectrophotometer provided with 1 cm matched quartz cells was used for absorbance measurements.

Reagents and materials

All chemicals were of analytical reagent grade and double distilled water was used to prepare solutions.

Ferric Chloride was purchased from Loba Cheme, Mumbai, India. Fehling solution was prepared by taking 2 mL of Fehling A solution and 2 mL of Fehing B solution (Blulux Laboratories, India) and diluted to 25 mL with water.

Standard drug solution

Pharmaceutical grade ofloxacin was kindly provided by Cipla Pvt. Ltd., India. A stock standard solution equivalent to 1mg/mL ofloxacin was prepared by dissolving 50 mg of pure drug in 0.1 M Hydrochloric Acid and diluting to 50 mL in calibrated flask with 0.1 M Hydrochloric Acid. Pharmaceutical grade cefixime was kindly provided by Cipla Pvt. Ltd., India. A stock standard solution equivalent to 1mg/mL cefixime was prepared by dissolving 50 mg of pure drug in 0.1 M Sodium Hydroxide and diluting to 50 mL in calibrated flask with 0.1 M Sodium Hydroxide.

Methods

Different aliquots (0.0, 1.0,.....4.0 mL) of 1 mg/mL ofloxacin solution were accurately measured and transferred into a series of 50 mL volumetric flasks. 1 mL of 1.0% aqueous solution of ferric chloride was added in each flask. All flasks were kept at room temperature for 5 minutes, and volume made up to the mark with 0.1 M HCl. Absorbance was measured at 435 nm *versus* reagent blank which shown nil absorbance against corresponding wavelength. The calibration curve was prepared to calculate the amount of drug. Different aliquots (0.0, 0.5,.....2.0 mL) of 1 mg/mL cefixime solution were accurately measured and transferred into a series of 50 mL volumetric flasks. 5 mL of Fehling solution was added in each flask. All flasks were kept at room temperature for 2 minutes, and volume made up to the mark with water. Absorbance was measured at 490 nm *versus* reagent blank which shown nil absorbance against corresponding wavelength. The calibration curve was prepared to calculate the amount of drug.

Assay of pharmaceutical formulations

Twenty tablets were weighed accurately and ground into a fine powder. Powder equivalent to 100mg of ofloxacin was weighed accurately and transferred into a 100 mL volumetric flask with 60 mL 0.1 M HCl. The content was shaken for 15-20 min, diluted to volume with 0.1 M HCl, and filtered using a Whatman No. 42 filter paper. First 10 mL portion of filtrate was discarded and subsequent portions were subjected to analysis. Again Powder equivalent to 100mg of cefixime was weighed accurately and transferred into a 100 mL volumetric flask with 60 mL 0.1 M NaOH. The content was shaken for 15-20 min, diluted to volume with 0.1 NaOH, and filtered using a Whatman No. 42 filter paper. First 10 mL portion of filtrate was discarded and subsequent portions were subjected to analysis.

RESULTS AND DISCUSSION

The optical characteristics such as absorption maxima, Beer's law limits, Molar absorptivity and Sandell's sensitivity for the methods are presented in Table 1.The regression analysis using the method of least squares was made for the slope and intercept obtained from different concentrations are summarized in Table 1. The precision and accuracy were found by analyzing six replicate samples containing known amounts of the drug and the results are summarized in Table 1

Table1: Optical characteristic, precision and accuracy of proposed method

Parameters	Ofloxacin	Cefixime
λ max (nm)	435	490
Beer's law limit (µg/ ml)	15-75	5-40
Sandell's Sensitivity (µg/cm^2)	0.034	0.033
Molar absorptivity (Litre.mole^{-1}.cm^{-1})	1.114 x 10^4	1.127 x 10^4
Regression equation (Y)		
Intercept (a)	0.2317	0.0048
Slope (b)	0.0045	0.0031
% RSD	1.020	1.67
% Range of errors (95% confidence limits):		
0.05 significance level	0.852	1.387
0.01 significance level	1.262	1.999

Y = bx + a, where Y is the absorbance and x is the concentration (for ofloxacin and cefixime) in µg/ mL for six measurements.

The accuracy of the above method was ascertained by comparing the results obtained with the proposed and reference methods in the case of formulation are presented in Table 2.

Table 2: Assay and recovery of ofloxacin and cefixime in pharmaceutical formulations

Formulation	Labeled amount (mg)		Recovery % (reference method)*		Recovery % (proposed methods) #	
	Ofloxacin	Cefixime	Ofloxacin	Cefixime	Ofloxacin	Cefixime
F1	200	200	99.62	99.58	99.70	99.49
F2	200	200	99.67	99.60	99.72	99.52

F1 and F2 are tablets from different batches (MAHACEF PLUS, Akums Drugs & Pharmaceuticals Ltd.)

* Reference method was UV method developed in the laboratory; # Recovery amount was the average of six determinants.

As an additional check on the accuracy of these methods, recovery experiments were performed by adding known amounts of pure drug to pre-analyzed formulation and percent recovery experiments were also done. Recovery experiments indicated the absence of interferences from the commonly encountered pharmaceutical additives and excipients.

CONCLUSION

It could be concluded that the developed method for estimation of cefixime and ofloxacin in same formulation is simple, sensitive, relatively precise and economical. The proposed methods are used for the routine analysis of the drugs in the quality control.

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