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RESEARCH ARTICLE

DEVELOPMENT AND VALIDATION OF FIRST ORDER DERIVATIVE SPECTROPHOTOMETRIC METHOD FOR ESTIMATION OF MOXONIDINE IN BULK DRUG AND PHARMACEUTICAL DOSAGE FORM

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Abstract:
A rapid, precise, accurate and specific first-order derivative spectrophotometric method was developed for the determination of moxonidine in pharmaceutical formulation. The technique was applied using 0.1M HCl as diluent. The first-order derivative spectra were obtained and determination was made at 232 nm. The method showed good linearity in the concentration range of 10-50 µg/ml with correlation coefficient 0.999. The recovery ranged between 99.50 and 100.50 %. The proposed method will be suitable for the analysis of moxonidine in bulk and in marketed preparations. The results of analysis have been validated statistically and by recovery studies.

Keywords: Moxonidine, First-order derivative, Method validation

Introduction

Moxonidine
Chemically moxonidine is 4-chloro-*N*-(4,5-dihydro-1*H*-imidazol-2-yl)-6-methoxy-2 methylpyrimidin-5-amine. Moxonidine is a new generation centrally acting antihypertensive drug licensed for the treatment of mild to moderate essential hypertension. It may have a role when thiazides, beta-blockers, ACE inhibitors and calcium channel blockers are not appropriate or have failed to control blood pressure. In addition, it demonstrates favourable effects on parameters of the insulin resistance syndrome, apparently independent of blood pressure reduction.

The literature review reveals few analytical methods such as HPLC[1-2], HPTLC[3], UV spectrophotmetric,GC-MS were reported for the estimation of moxonidine This paper reports the development and validation of a first order derivative spectrophotometric method for the determination of moxonidine in pharmaceutical formulation

MATERIALS AND METHODS:

Apparatus:

Instrumentation and Materials
A U.V. visible double beam spectrophotometer shimadzu 1601 UV-Visible Spectrophotometer with 1cm U.V. matched quartz cells were used.

Chemicals and materials:
Reference standard of moxonidine was provided by Macleods Pharmaceuticals.Tablet formulations were procured from a local pharmacy.

Preparation of stock solution of moxonidine
Standard moxonidine (10 mg) was accurately weighed and transferred to 10 ml volumetric flask. It was dissolved properly and diluted up to the mark with 0.1MHCl to obtain concentration of 1000 µg/ml. This solution was further diluted to obtain concentration of 10 µg/ml which was used as working standard solution.

Spectrophotometric measurements

The absorbance of the solutions containing moxonidine at 10 µg/ml was determined in the UV range 200-400 nm (Fig. 1) using an appropriate blank. The first order derivative spectra were obtained by instrumental electronic differentiation in the range of 200-400 nm. The amplitude values obtained in the first-order derivative spectra at 232 nm was selected for the analysis of moxonidine.

VALIDATION OF METHOD PARAMETERS:

Specificity
The specificity of the method was evaluated through the analysis of a market sample solution, which contains number of pharmaceutical excipients in their usual concentration

Linearity
The stock solution solutions of moxonidine were diluted accordingly to get a final concentration ranging from 10,20,30,40 and 50 µg/ml. Each solution was prepared in triplicate. The linearity was evaluated by linear regression analysis, which was calculated by the least square regression method.

Accuracy
Accuracy of proposed method from excipients was determined by recovery experiments. Recovery experiments were carried out in three levels of concentration. The amounts of standard recovered were calculated in the terms % recovery.

Assay of tablet sample

A 100 mg equivalent of moxonidine tablet powder was transferred into a 100 ml volumetric flask containing 50 ml of 0.1MHCl and sonicated for 15 minutes. The volume was completed with the same. The solution was filtered through Whatmann filter paper and diluted accordingly to obtain 10 µg/ml concentrations. The amplitude value of the solution was measured at 232nm. Concentration of sample solution was calculated from amplitude value of standard solution of 10 µg/ml prepared simultaneously

RESULTS AND DISCUSSION:

Linearity
Linearity was evaluated by calculation of correlation coefficient. The R^2 value was found to be 0.999 and the values are given in Table 1 and in Fig 2-3.

Accuracy
The mean absolute recovery of moxonidine is between 99.50- 100.50%, the values were given in Table 2

Precision

By the precision studies (system precision and method precision) the relative standard deviation values were obtained as less than 2%, the values were given in Table 3 and 4.

Assay:

By performing assay the amount of moxonidine present in tablets was estimated and the results are shown in Table 5.

CONCLUSION:

The first-order derivative spectrophotometric method is rapid, accurate and precise. The results of the analysis of the tablets by this method were reproducible reliable, and in good agreement with labeled claim of the drug. There was no interference from the excipients present in the tablets, hence the proposed method can be used in the estimation of moxonidine in bulk and pharmaceutical dosage forms in a routine manner.

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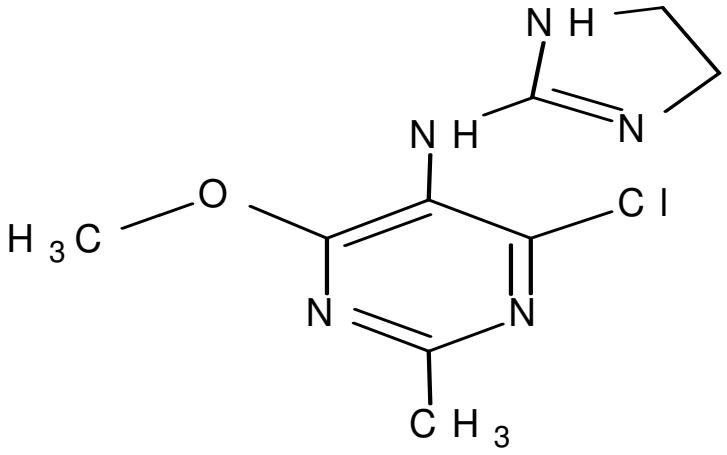

Figure – 1 Chemical structure of moxonidine

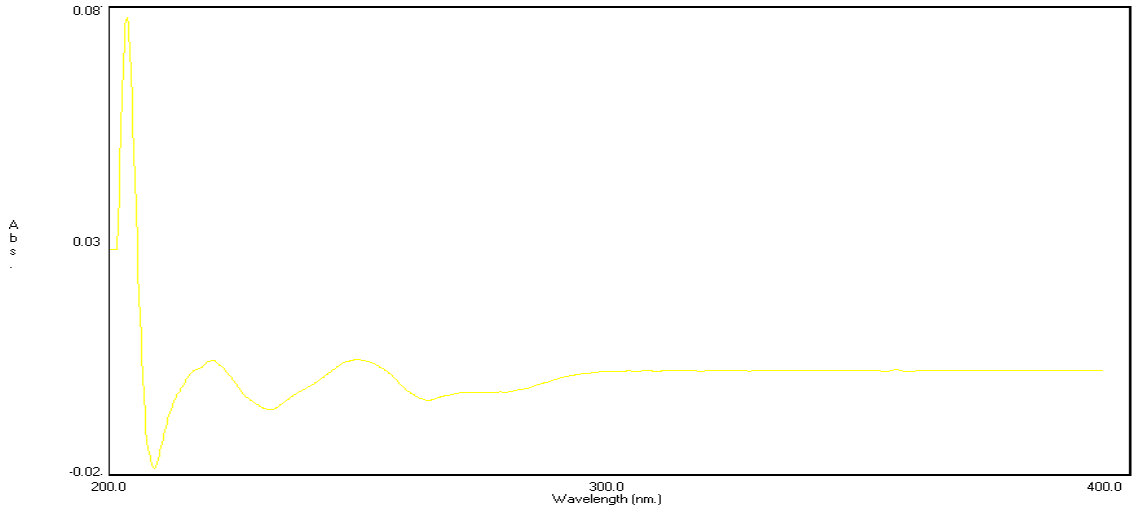

Figure – 2 Overlay spectrum of moxonidine

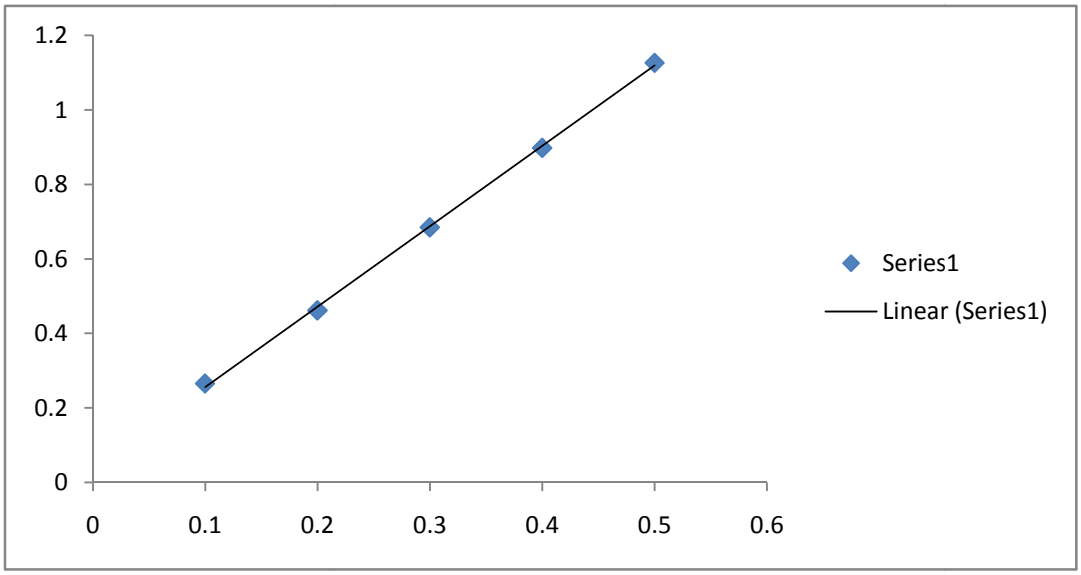

Figure – 3 Linearity plot of moxonidine

Table – 1: System suitability parameters

Parameters	Observations
Wave length measured (nm)	232
Linearity range (µg/ml)	10-50
Correlation Coefficient	0.999

Table 2. Results of recovery studies

Sr. No.	Amount of drug (mg)	Amount found (mg)	% Recovery
1	8	7.99	99.66
2	10	10.01	99.93
3	12	11.99	99.99

Table – 3 System Precision of Moxonidine

Sr. No	Concentration (mcg/ml)	Standard absorbance
1	20	.468
2	20	.470
3	20	.471
4	20	.465
5	20	.468
	Average	.4684
	SD	.0024
	%RSD	.512

Table – 4 Method Precision of moxonidine

Set	Label claim(mg)	Amount estimated(mg)	% Assay
1	2	1.98	99.00
2	2	2.01	100.5
3	2	2.01	100.5
4	2	1.98	99.00
5	2	1.99	99.50
		Average	99.70
		SD	.758
		%RSD	.760

Table – 5 Assay of moxonidine

Set	Label claim(mg)	Amount estimated(mg)	% Assay
1	2	1.99	99.50
2	2	2.01	100.5
3	2	2.01	100.5
		Average	100.16
		SD	.577
		%RSD	.576