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Research Article

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Spectrophotometric determination of silodosin in pharmaceutical formulations by charge transfer complex method

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ABSTRACT

Highly sensitive, simple, and economical UV-spectrophotometric method has been developed for the determination of silodosin in bulk and dosage forms. The proposed method was based on the charge transfer reaction of selected drug as n-electron donors with acceptor 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) to form red color charge-transfer complexes. This reaction is instantaneous and quantitative. Silodosin has the maximum absorbance at 410 nm and Beer's law limit was obeyed at 30-120 µg/ml. The optical characteristics of the proposed method such as molar absorptive, sand ell's sensitivity, slope and intercept were 1.1254 L.mole^{-1}cm^{-1}, 0.0033 µg.cm^{-2}, 0.0040 and 0.0014 for silodosin respectively. The developed method was found to be simple, specific, robust, accurate and precise for the determination of silodosin.

Key words: Silodosin, chloroform, methanol, DDQ and UV-Spectrophotometric method.

INTRODUCTION

Silodosin is chemically known as 1-(3-Hydroxypropyl)-5-[(2R)-2-({2-[2-(2, 2, 2-trifluoroethoxy) phenoxy] ethyl} amino) propyl]-2, 3-dihydro-1*H*-indole-7-carboxamide [1, 2]. Molecular formula is $C_{25}H_{32}F_3N_3O_4$ with a molecular weight of 495.53.

Silodosin[1] is a selective antagonist of treatment benign prostatic hyperplasia. It relieves the symptoms of benign prostatic hyperplasia alfa-1-adrenoreceptors and a class of medications called alpha-blockers [3, 4]. It is used for the symptomatic by relaxing the muscles of the bladder and prostate. Silodosin has an apparent volume of distribution of 49.5 L and is approximately 97% protein bound. Oral administration of ^{14}C-labeled silodosin, the recovery of radioactivity after 10 days was approximately 33.5% in urine and 54.9% in feces. After intravenous administration, the plasma clearance of silodosin was approximately 10 L/hour [5, 6].

Literature survey reveals that very few analytical methods are available for the determination of silodosin. UV Spectrophotometric [7, 8] method, one HPLC [9], Volta metric techniques [10] and human plasma by using liquid chromatography-tandem mass spectrometry [11] methods are available.

EXPERIMENTAL SECTION

2.1 Instrumentation

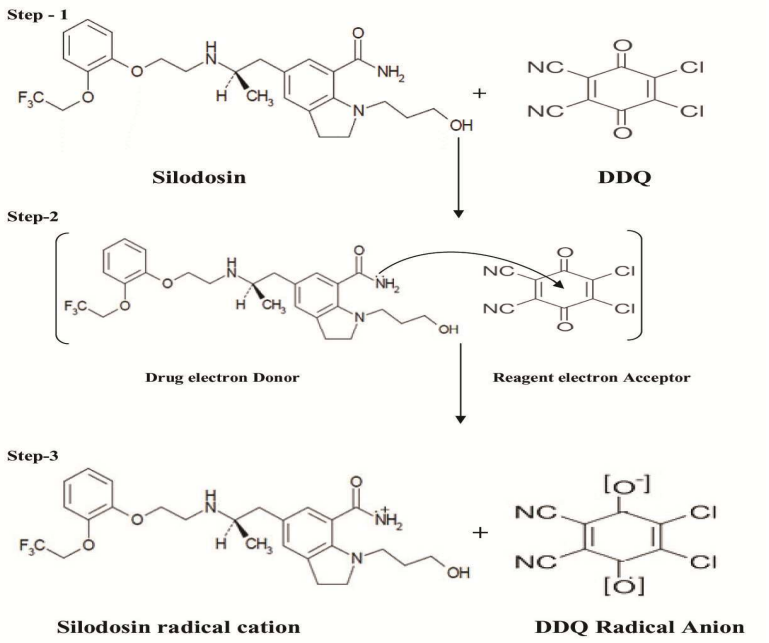
A Shimadzu UV-visible double beam spectrophotometer (model 2450) with 1 cm matched quartz cells was used for the spectral measurements.

2.2 Chemicals and reagents
All the chemicals used were of analytical grade. Double distilled water was used for all the experimental studies.

2.3 DDQ solution (1% w/v)
DDQ (2, 3-dichloro5, 6-dicyano-p-benzoqunone) (Loba Chem., India) solution is prepared by dissolving 100 mg in 100 ml of distilled water.

2.4 Silodosin solution
The stock solution (mg ml^{-1}) of silodosin is prepared by dissolving 50 mg of drug in 50 ml of methanol. A portion of this stock solution is diluted stepwise with the methanol to obtain the working standard solutions of 100 µg/ml.

2.5 Spectrum of silodosin treated with DDQ
The wavelength of maximum absorbance of the silodosin drug treated with DDQ solution is ascertained by the following procedure. A 1.0 ml of silodosin solution (100 µg/ml) is transferred into standard flask. To this solution, 0.1 ml DDQ reagent is added to form a red colored solution. The final volume is brought to 10 ml with methanol. The resultant solution is well mixed and allowed to stand for 5 minutes for completion of the reaction. The absorbance of the red colored solution is measured in the wavelength range of 400 to 600 nm, against the reagent blank. The spectrum is given in fig.1.From figure it is clear that silodosin drug treated with DDQ solution has maximum absorbance at 410 nm.



Scheme: 1The reaction sequence of charge transfer complex method

2.6 Assay procedure
The fresh aliquots of standard drug solution of silodosin ranging from 0.2-1.2 ml were transferred into a series of 10 ml volumetric flasks. To each flask, 1.0 ml of DDQ solution was added and kept on water bath for 20 minutes for complete color development and cooled and transferred the colored solution in to 100 ml separating funnel. The mixture was extracted twice with 10 ml chloroform by shaking for 2 minutes and then allowed to stand for clear separation of the two phases. The absorbance of the separated chloroform layer, i.e. reddish colored complex was measured at 410 nm against the reagent blank. Calibration graph was obtained by plotting absorbance values against the concentration of silodosin solution. The calibration curve is found to be linear over a concentration range of 30-120 µg/ml of silodosin.

2.7Assay of silodosin in pharmaceutical formulations

The method is then applied for the determination of the drug from the marketed tablet formulations. Tablets are weighed and contents are powdered and well mixed. The powder equivalent to 50 mg of silodosin is dissolved in methanol, filtered and residue is washed with distilled water and the volume is made up to 50 ml with methanol. Further, more over dilution is made as described in the preparation of standard solution of silodosin. Further analysis is carried out as per procedure described above and results are summarized in the Table 2. The amount of drug present in the sample is estimated from calibration graph.

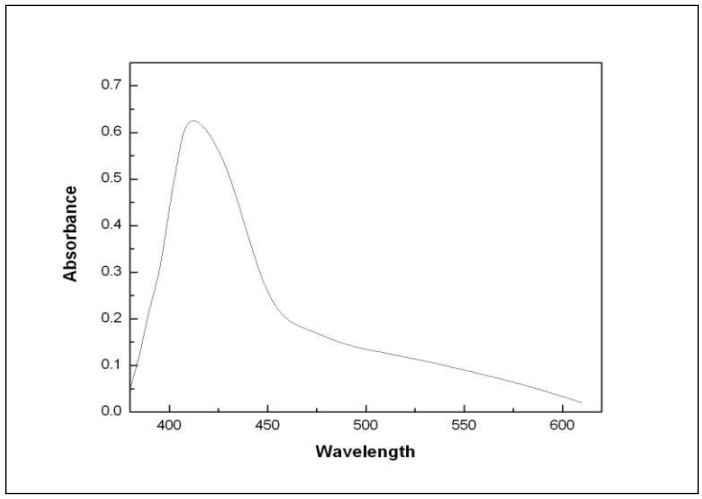


Fig: 1 spectrum of silodosin drug treated with DDQ solution

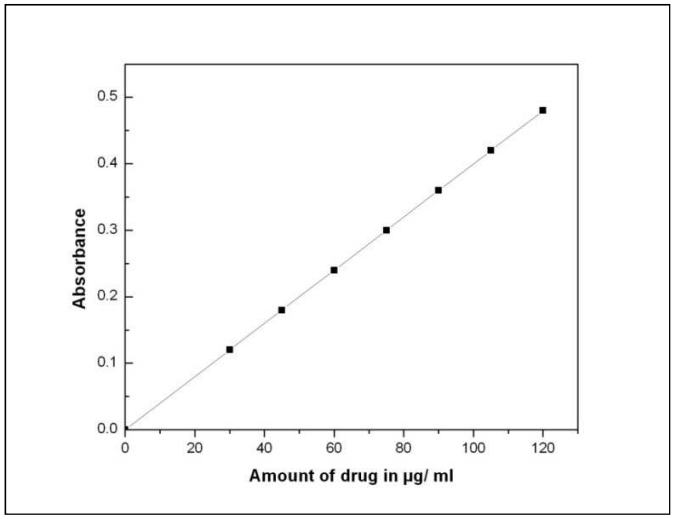


Fig: 2 calibration curve of silodosin

RESULTS AND DISCUSSION

The optical characteristics such as Beer's law limits, Sandal's sensitivity, molar extinction coefficient, percent relative standard deviation, percent range of percent relative standard deviation and percent range of error (0.05 and 0.01 confidence limits) were calculated for the method and results are summarized in table 1. The values obtained for the determination of silodosin in pharmaceutical formulations (Tablets) by the proposed method are presented in table 2. The studies reveal that the recipients and other additives usually present in the tablets did not interfere with the results obtained in the proposed method.

Table 1 Optical characteristics of the Proposed Methods

Parameters	Proposed method
λ max(nm)	410
Beer's law limit(µg/ml)	30-120
Molar absorptivity (L.mole^{-1} cm^{-1})	1.1254
Sandal's sensitivity (µg.cm^{-2}/0.001 A.U)	0.0033
Slope(b)	0.0040
Intercept(a)	0.0014
Correlation coefficient(r^2)	0.9988
Relative standard deviation (RSD)%	0.3333
LOD(µg/ml)	0.7446
LOQ(µg/ml)	2.4797
Color	Reddish pink

Table 2 Assay of Silodosin in pharmaceutical formulations

Tablets	Label led amount mg/ml	Amount found mg/ml	%Recovery	±SD	% RSD
Rogitine	250	249.53	99.33	0.0577	0.2854
Regitine	250	249.53	99.33	0.0288	0.2906
Or averse	250	249.43	99.64	0.0450	0.3016

**Average of five determination based on label claim*

CONCLUSION

The proposed method is found to be simple, precise, accurate, time saving, reproducible and can be conveniently adopted for routine analysis of estimation of silodosin in bulk drug samples and pharmaceutical formulations

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