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Research Article

Differential spectrophotometric method for estimation and validation of Verapamil in Tablet dosage form

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Abstract

The aimed of current research to development of the simple, rapid and sensitive Differential spectrophotometric method for the estimation of Verapamil in tablet dosage form. In this method two medium was use acid and alkaline and the difference spectrum was calculated. 0.1N HCL and 0.1N NaOH was used in this differential method. The λmax 278, Beers law limits 5-25µg/ml, regression equation Y= 0.024x-0.009, slope 0.024, intercept 0.09, correlation coefficient (r^2) 0.998, %RSD <1.5, % Recovery (Tablet) 100.46% was shows the good efficacy and results. This method future scope in quality control of the verapamil in simple, precise and economically and it recommended for the routine drug quality analysis investigation.

Keywords: Difference spectrophotometry, Verapamil, Calibration, Tablet, Validation.

INTRODUCTION

Verapamil Hydrochloride is a chemically known as 5-[N-(3,4-dimethoxy-phenethyl)-N-methyl-amino]-2-(3,4-dimethoxyphenyl)-2-isopropylvaleronitrile hydrochloride. This is a calcium channel blocker, it show the anti-arrhythmic drug to manage supra ventricular arrhythmias and it also anti-anginal drug. Some analytical methods for quantitative determination of Verapamil are described viz., capillary electrophoresis, tandem mass spectroscopy detection (LC-MS/MS), HPLC and inverse voltampere method. By the various survey of literature revealed that oxidative method of quantification of these drugs by Ce (IV) have been not reported yet, although the method simple sensitive, precise and accurate[1].

HPLC was one of the methods of choice of researcher for the analysis of the drug showing an overlapped spectrum on UV spectrophotometry. But HPLC being an expensive and also the time consuming method from the last few years, chemometric spectrophotometry is immensely attracting the analysts and play an importance role in accurate detection of complex drug mixtue[3].

Verapamil was introduced in 1962 as coronary vasodilator and it also prototype of the Ca^{2+} antagonists used in cardiovascular disease. Verapamil major effect is on the slow calcium channel. The inhibition of the action potential inhibits one limb of the reentry circuit believed point. It is class IV antiarrhythmic drug. Verapamil also cause a change in Hemodynamic preload. The drugs reduce systemic vascular resistance and mean blood pressure with minor effect on cardiac output[5].

Differential spectrophotometry is an analytical technique which has been used to improve the selectivity and accuracy of the measurement. It is based on measuring the difference of two equimolar solution of the analyte in different chemical forms. It is not only eliminates matrix interference due to excipients, but it also resolves spectral overlap of other accompanying drugs[7].

The accuracy and selectivity of conventional UV absorption method is also increased by conversion of normal zero-order or differential UV spectra into higher order. So the application of differential spectrophotometry is an expected to have the advantages of both derivative spectrometry like first and second combined with delta spectrophotimetry[9]. The aim of current study to developed differential spectrophotometric method for the estimation of verapamil in tablet dosage form. The accuracy, precision, %RSD and recovery study was indicated the reproducibility.

MATERIALS AND METHOD

Chemicals and equipments

Hydrochloric acid, Sodium hydroxide, pure Verapamil (Nicholas Piramal), Verapamil (Veramil 40mg) tablet purchase from Themis Medicare.

Jasco UV-Visible double beam Spectrophotometer with 1 cm matched pair quartz cell and spectral bandwidth of 2cm.

EXPERIMENTAL

1. METHODS[10]

Selection of solvent

0.1N HCL and 0.1N NaOH was selected as solvent for developing spectral characteristics of drug. The selection was made after the trial of different acids and bases and their different normality.

Preparation of Standard drug solution

A. In HCL

An accurately 10mg of Verapamil weighed and transfer into 100ml volumetric flask, dissolved in 0.1N HCL and volume was made up to produced 100µg/ml solution.

B. In NaOH

An accurate 10mg of verapamil weighed and transfer into 100ml volumetric flask, dissolved in 0.1N NaOH and volume was made up to the produced 100 µg/ml solution.

For A and B the separate preparation of different concentration, aliquots of stock solution were transferred into a series of 10ml standard volumetric flask and volume were adjusted with respective solvent of 5 different concentrations from 5 to 25 µg/ml of Verapamil were prepared.

Wavelength selection

Standard solutions of the drug were scanned to generate an absorption spectrum. The Wavelength at which drug show maximum absorption was selected as λmax and used as a analytical wavelength. All the dilutions were scanned in the range 200-400 nm,

The wavelength selected for estimation of verapamil was 278nm (Fig 1& 2)

Plotting Calibration curve

The calibration curve of verapamil was done in the range of concentration 5 to 25 µg/ml. (Table 1 & Fig 3)

Assay of verapamil in tablet[11]

Marketed formulations of containing 5mg of verapamil were analyzed by this method. 20 tablets were triturate an amount equivalent to 10mg of verapamil was weighed and transfer into the 100ml volumetric flask. The content of the flask were dissolved in the 50ml of the 0.1N HCL and 0.1N NaOH separately with the aid of ultra-sonication for 10min. The solution was filtered through whatman filter paper no. 41 and then final volume of the solution was made up to 100ml with same solvents to get a stock solution containing 100 µg/ml of verapamil in 0.1N HCL and 0.1N NaOH. After appropriate dilutions, the absorbance were measured and the concentration of each analyte was determined with the equation obtained from calibration curve.(Table 2) shows the λmax, %RSD, intraday, inter-day and % recovery of tablet was noted.

2. VALIDATION[12]

The proposed differential UV spectrophotometric method was statistically validated for linearity, accuracy, precision, repeatability, reproducibility, robustness, range, sensitivity, limit of detection and quantification. (Table 2) shows the beers law limits, regression equation, slope, intercept, correlation coefficient (r^2) was calculated.

Linearity

Linearity of the proposed method was by the plotting 5 point calibration curve. The result used to calculate % relative standard deviation using the linear regression equation of the line by least square regression method.(Fig 3) good result was calculated.

Recovery study

Accuracy and sensitivity of analysis was determine by performing recovery study by spiking different concentrations of pure drug in the reanalyzed tablet sample.(Table 2) show the results. The recovery was 100.46% was observed.

RESULTS AND DISCUSSION

Spectral Study / Wavelength selection

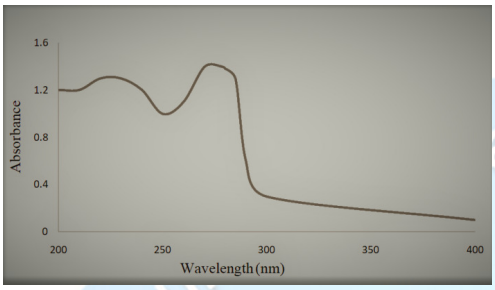

Fig 1: Spectra of Verapamil in 0.1N HCL

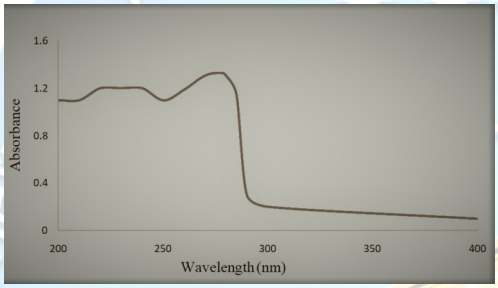

Fig 2: Spectra of Verapamil in 0.1N NaOH

From the Fig 1 & Fig 2 the acidic and alkaline medium results the wavelength was selected 278nm.

Calibration plot

Table 1: Concentration and Absorbance of Verapamil in Acidic and Alkaline medium (Amplitude)

Concentration	Absorbance		Amplitude
(µg/ml)	0.1N HCL	0.1N NaOH	(Difference)
5	0.357	0.258	0.099
10	0.711	0.476	0.235
15	1.012	0.660	0.352
20	1.335	0.850	0.485
25	1.561	0.966	0.595

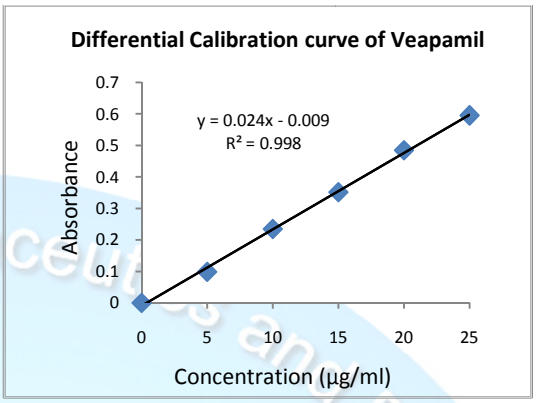

Fig 3: The difference Absorption Calibration Curve of Verapamil in Acidic and Alkaline solution.

Validation of method and regression analysis

Table 2: regression analysis and method validation

Parameters	Differential spectroscopy
λ max, nm	278
Beer's law limits (µg/ml)	5-25
Regression equation (Y*)	Y=0.024x-0.009
Slope (b)	0.024
Intercept (a)	0.09
Correlation coefficient(r^2)	0.998
%RSD**	<1.5%
Intraday %	1.1-1.8
Inter-day %	1.1-1.9
% Recovery (Tablet)	100.46%

CONCLUSION

The research indicates the method is found to be simple, precise. It had good reproducibility, selective and sensitive method. The statistical parameters clearly indicate the reproducible and accuracy of the method by spiking the different concentrations of pure verapamil. This method recommended for routine and quality control analysis of the investigated drug in tablets.

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