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DEVELOPMENT AND VALIDATION OF UV SPECTROPHOTOMETRIC METHOD FOR THE ESTIMATION OF OMEPRAZOLE IN BULK AND PHARMACUTICAL FORMULATION

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ABSTRACT

To develop simple, economical, precise and less time consuming UV method for the estimation of Omeprazole in bulk and pharmaceutical formulations. The method is based on UV spectroscopic technique. Omeprazole shows the maximum absorbance at 301nm in absorption maxima method. Drug followed the linearity in the range of 5-25µg/ml for this method with correlation coefficient (r^2) of 0.999. The results of analysis have been validated statistically and recovery studies confirmed the accuracy of the proposed method. The method was validated as per the International Conference on Harmonization (ICH) guidelines. The proposed method is recommended for routine analysis since it is rapid, simple, accurate and sensitive.

Keywords: Omeprazole, HPLC, UV spectrophotometry, absorption maxima method.

INTRODUCTION

Omeprazole [1-2] is chemically named as 6-methoxy-2-{[(4-methoxy-3,5-dimethylpyridin-2-yl)methane]sulfinyl}-1H-1,3-benzodiazole, is official in IP, BP and USP[3-5]. It is a proton pump inhibitor used in the treatment of dyspepsia, peptic ulcer disease (PUD), gastroesophageal reflux disease (GORD/GERD), laryngopharyngeal reflux (LPR) and Zollinger–Ellison syndrome. It works by decreasing the amount of acid made in the stomach [6]. Literature review shows that there are developed methods including spectrophotometric, HPLC and HPTLC methods for the estimation of Omeprazole alone and in combination of other drugs like Ondansetron, Domperidone etc.There are developed Spectrophotometric methods [7-11] of analysis in single or in combination. Omeprazole shows absorption in UV-visible range in Water was measured in absorption maxima method. In the present investigation simple and sensitive UV spectrophotometric method have been developed for the quantitative estimation of Omeprazole in bulk and its marketed formulations with good accuracy and economy. The structure of Omeprazole is shown in (Figure 1).

EXPERIMENTAL WORK

All the chemicals used during the experimental work are of Analytical grade. A Shimadzu UV-1800 UV/VIS Spectrophotometer was used with 1cm matched quartz cells. Tablets of OMETAB- 10mg were procured from local market.

Preparation of standard solution: The pure drug of about 10 mg was weighed and transferred in to a 10ml volumetric flask. The drug was dissolved completely in a few ml of Methanol and made up to the final volume with Methanol to get a stock solution of concentration 1000µg/ml. Aliquots of standard stock solution were pipette out and diluted suitably with water to get the final concentration of standard solutions.

Absorption maxima method: The solutions were scanned in the range of 400-200 nm against water as reference, and the peaks were observed in the spectra

at 301nm. The wavelength selected for analysis of drug was 301nm (Figure 2). The drug obeys the lamberts law in the range of 5-25 µg/ml. By using linearity plot (Figure 3) the quantification was carried out.

Optical characteristics: Optical characteristics such as Beer's law limit (µg/mL)**,** Correlation coefficient, Regression equation, Slope (m), and Intercept (c) were calculated (Table-1).

Analysis of tablet formulation: For the estimation of Omeprazole in pharmaceutical formulation by above method, 10 tablets of OMETAB-10mg brand were weighed and triturated to a fine powder. Tablet powder equivalent to 10mg was weighed and transferred to 100ml volumetric flask and dissolved in few ml of Methanol with the aid of ultra-sonication for 15min; this was filtered through whatman filter paper no. 41 to get the stock solution of 100 µg/ml various dilutions were prepared from tablet solution and analyzed for six times and the concentration for both the methods was calculated by using calibration curve (Table-2).

VALIDATION OF ANALYTICAL METHOD

The analytical method was validated according to ICH validation parameters [12].

Linearity: Fresh aliquots were prepared from standard stock solution ranging from 5-25 µg/ml and the absorbance values of each concentration was recorded at 301nm for this method using water as blank. The drug shows linearity between 5-25 µg/ml for this method (Table-3).

Precision: In intraday study, concentration of replicates of drug was calculated on the same day for three times. In inter-day study the concentration of drug were calculated on three successive days which expresses the laboratory variation in different days. In both intra and inter day precision study for the methods %RSD was calculated (Table-4(a), 4(b).

Accuracy: Accuracy of the developed method was confirmed by performing recovery studies at three different concentration ranges 80%, 100%, 120% each one in triplicate (Table-3). From the recovery studies it was clear that the method is very accurate for quantitative estimation of tablet as the statistical results were within the acceptance range (Table-5).

Limit of Detection and Limit of Quantification: The limit of detection and limit of quantification of Omeprazole by proposed methods were determined using calibration graphs. LOQ and LOD were calculated as

LOD = 3.3 X S.D/S

LOQ = 10 X S.D/S

Where S is the slope of the calibration curve and SD is the standard deviation of response of least concentration of calibration curve in three replicates.(Table-6).

Robustness: Robustness of the method was determined by carrying out the analysis at five different wavelengths (±2nm). The respective absorbance was noted and the result was indicated by % RSD (Table-7).

Ruggedness: Ruggedness of the method was determined by carrying out the analysis by two different analysts and the respective absorbance was noted. The result was indicated by % RSD (Table-7).

RESULTS AND DISCUSSION

The developed method was found to be precise as the %RSD values for intra-day and inter-day were found to be less than 2%. Good recoveries (98.75% to 100.41%) of the drug were obtained at each added concentration, which indicates that the method was accurate. The LOD and LOQ were found to be in sub-microgram level, which indicates the sensitivity of the method. The method was also found to be robust and rugged as indicated by the %RSD values which are less than 2%. The results of assay show that the amount of drug was in good agreement with the label claim of the formulation as indicated by % recovery (101.25%).

CONCLUSION

The proposed methods are simple, sensitive, and cost-effective. Validated in terms of precision, linearity and accuracy. The results are reproducible, and can be used successfully for the estimation of Omeprazole in bulk and its pharmaceutical formulations.

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Table1: Optical characteristics

Optical characteristics	Method A
Beer's law limit (µg/ml)	5-25
Correlation coefficient (r^2)	0.999
Regression equation	y = 0.032x + 0.001
Slope (a)	0.032
Intercept (b)	0.001
LOD	0.157µg/ml
LOQ	0.447µg/ml

Table 2: Analysis of Formulation

DRUG	LABEL CLAIM (mg/ Tablet)	AMOUNT* FOUND (mg/ Tablet)	% AMOUNT FOUND	%RSD
OMEPRAZOLE(OMETAB)	10	10.05	101.25	0.012

*Mean of three readings

Table3: Linearity of Omeprazole

S.no	Concentration in µg/ml	Absorbance UV Method
1	5	0.160
2	10	0.344
3	15	0.484
4	20	0.664
5	25	0.820

Table4 (a): Intra-day precision:

S.no	Conc (µg/ml)	Absorbance			%RSD
		Morning*	A.noon*	Evening*	
1.	10	0.344	0.342	0.342	0.33

Table4 (b): Inter-day precision:

S.no	Conc (µg/ml)	Absorbance			%RSD
		Day1*	**Day2***	**Day3***	
1.	10	0.344	0.342	0.339	0.73

*Mean of six replicates

Table5: Accuracy studies of Omeprazole

Method	Amount of µg/ml Tablet	Pure drug	% of drug added	Amount recovered	%recovered	% RSD
	10.0	8	80	7.98	99.75	
UV	10.0	10	100	9.95	99.50	0.47
	10.0	12	120	12.05	100.41	

Table6: LOD and LOQ of Omeprazole

Standard	LOD(µg/ml)	LOQ(µg/ml)
OMEPRAZOLE	0.157	0.477

Table7: Robustness and Ruggedness of Omeprazole

Parameter		%RSD
Robustness	Change in λmax **(**± 2 nm**)**	0.62
Ruggedness	1st analyst	0.86.
	2nd analyst	1.2

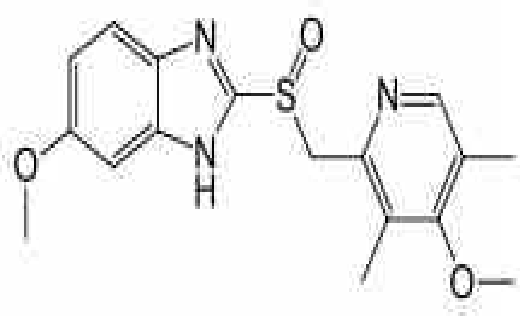

Figure1: Structure of Omeprazole

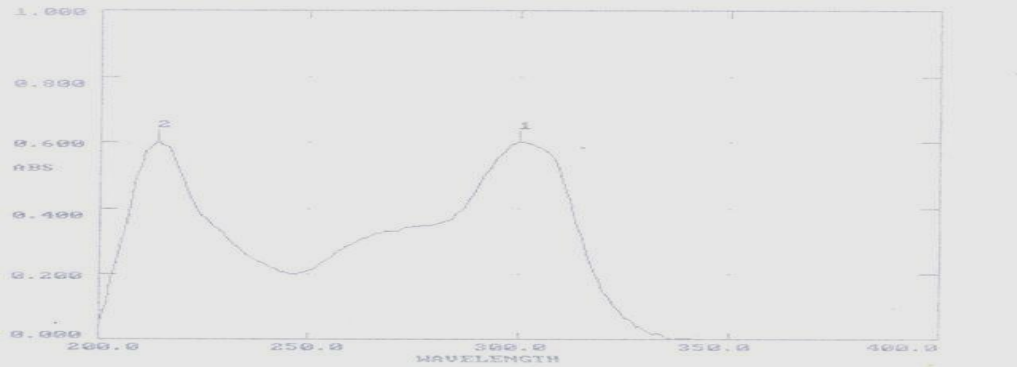

Figure2: Absorption maxima spectrum of Omeprazole

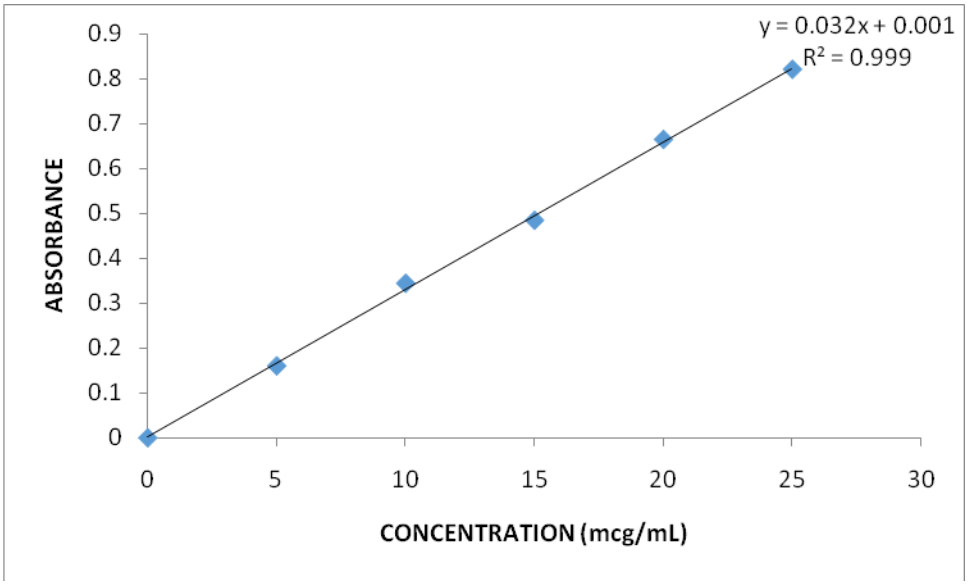

Figure3: Calibration curve of Omeprazole at 301nm

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