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Research Article

DEVELOPMENT AND VALIDATION OF ANALYTICAL METHOD FOR ESTIMATION OF PREDNISOLONE IN BULK AND TABLETS USING UV-VISIBLE SPECTROSCOPY

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ABSTRACT

Prednisolone is used as anti-inflammatory or immune suppressive agent. Various methods for analysis of the same are available but are time consuming and expensive. Here we have developed two new, precise and simple UV spectrophotometric methods for estimation of Prednisolone from tablet formulation. The drug obeyed the Beer's law and showed good correlation. Absorption maxima of Prednisolone in methanol were found to be at 244 nm. Beer's law was obeyed in concentration range 2– 12 mcg/ml. The absorbance was found to increase linearly with increasing concentration of prednisolone, which is corroborated by the calculated correlation coefficient value of 0.9995 (n=6).The results of analysis were validated by recovery studies. The recovery was more than 98%. The method was found to be simple, accurate, precise and economical.

Keywords: Prednisolone, UV Spectrophotometric, AUC, Linearity.

INTRODUCTION

Chemically Prednisolone is a glucocorticoid and its IUPAC name is (8S,9S,10R,11S,13S,14S,17R)-11,17-dihydroxy-17-(2-hydroxyacetyl)-10,13 dimethy l7,8,9,11,12,14,15,16-octahydro-6H cyclopenta[a]phenanthren-3-one. Prednisolone is used as anti-inflammatory or immune suppressive agent and it is official in India Pharmacopoeia[1]. In our Literature survey revels that for Prednisolone HPLC[2,3,] Spectrophotometric[4-6] and solid phase extraction[7] and HPTLC[8] methods have been reported for its determination in commercial formulation. However some of these methods are costlier and time consuming. To overcome these difficulties Spectrophotometric analysis serves to be the quickest, promising and reliable method for routine analytical needs. The aim of the present study is to develop two new simple, rapid, reliable and precise UV Spectrophotometric methods for analysis of Prednisolone from tablet formulation; first method is based on measurement of UV absorbance of Prednisolone at 244 nm in methanol. Second method involves the calculation of integrated value of absorbance with respect to the wavelength between two selected wavelength 241 nm and 247 nm. Area calculation processing item calculates the area bound by the curve.

MATERIALS AND METHODS

Chemicals

Gift samples of Prednisolone were provided by fourrts(India). Methanol (Qualigens laboratory, Mumbai), All solutions were prepared daily.

Instrumentation and analytical conditions

The UV method was performed on a Double-beam Shimadzu UV-Visible spectrophotometer, 1700, with spectral bandwidth of 2 nm, wavelength accuracy ± 0.5 nm and a pair of 1-cm matched quartz cells was used to measure absorbance of solution. Working wavelength for UV method was 244nm of Prednisolone.

Preparation of Standard Solutions

A stock solution containing 100mcg/ml of pure drug was prepared by dissolving accurately weighed 10mg of Prednisolone in methanol and volume was adjusted to 100ml with the same in 100ml volumetric flask.

Linearity and Calibration

The aliquots working standard solution was diluted serially with methanol to obtain the concentration range of 2 – 12 mcg/ml. A calibration curve for Prednisolone was obtained by measuring the absorbance at the λmax of 244 nm (for method I) and by measuring area under curve between 241 to 247 (for method II). Statistical parameters like the slope, intercept, coefficient of correlation, standard deviation, relative standard deviation, and standard error were determined.

Analysis of marketed tablet formulation

Accurately weighed the 20 tablets and powdered. The powder equivalent to 5mg of Prednisolone was transferred to 100ml volumetric flask and 20ml methanol is added to dissolve the Prednisolone in it and made the volume to mark with the same. This

Mixture was sonicated for 10 minutes and filtered through Whatmann filter paper No. 41. Aliquots (0.1ml, six times) of the sample were removed and diluted to 10 ml with methanol to obtain strengths as 10mcg/ml six time and determined the respective absorbance at 244 nm and area under curve between 241 nm to 247 nm against the methanol as blank.

Recovery Studies

Recovery studies were performed to judge the accuracy of the method.1ml of standard formulation (10mcg/ml) was taken in three 10ml volumetric flask and to it 80%, 100% and 120% (i.e. 0.8ml, 1.0ml, 1.2ml) of working standard solution (100mcg/ml) added respectively and made the volume up to the mark. The respective absorbance at 244 nm and area under curve between 241 nm to 247 nm was recorded against the blank. The amount of added concentration was determined from the obtained absorbance values and percent recovery was determined for the formulation.

RESULTS

The UV scan of standard solution between 200 – 400 nm showed the absorption maxima at 244 nm, shown in (fig.1). The Beer's law was verified from the calibration curve by plotting graphs of concentration vs absorbance (method I) and concentration vs area under curve (method II). Regression analysis showed very good correlation. The calibration plots revealed zero intercept which is clear by the regression analysis equation Y = 0.0642X + C. (Where Y is absorbance, m is the slope and X is the concentration of Prednisolone in mcg/ml) as obtained by the least square method. The results thus obtained are depicted in (Table No.1). The results of analysis for assay and recovery studies for two different formulations were studied and are shown in (Table No.2). No significant variations were observed on interday and intraday analysis.

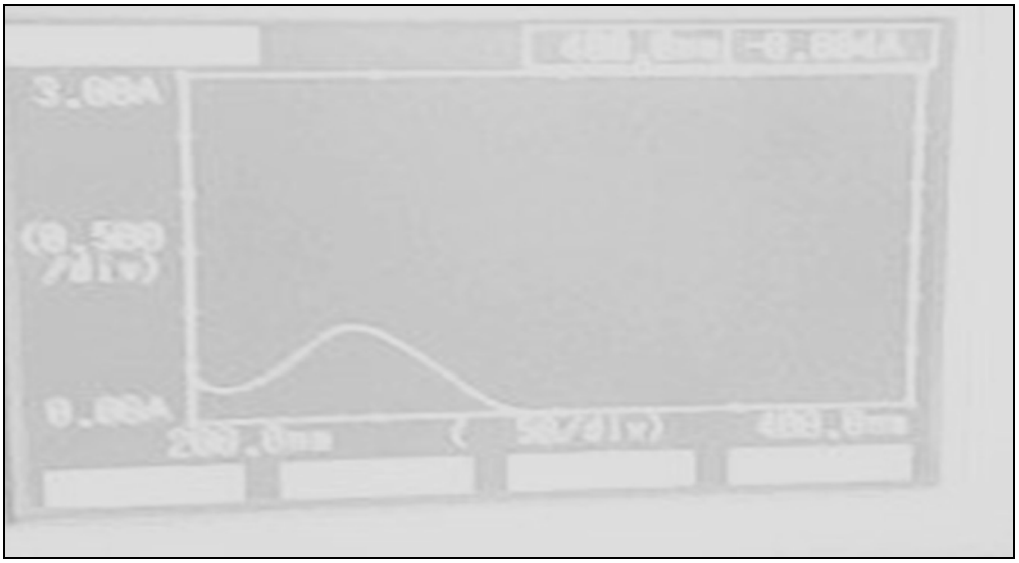

Fig. 1: Overlain spectra of Prednisolone in methanol

Table 1: Optical characteristics of Prednisolone

Methods	I	II
Absorption maxima	244	241-247
Beer's law limit	2 -12mcg /ml	2 -12mcg/ml
Coefficient of Correlation	0.9995	0.9990
Regression equation	0.9989	0.9985
Slope	0.0642	0.0631
y intercept	0.0352	0.0367

Table 2: Results of analysis of tablet and recovery study

Method	Formulation	Label claim	%Label Claim	Standard Deviation	Coefficient of variation	%Recovery Studies
I	Wysolone	5mg	99.998	0.69043	0.00612	100.36±0.135
II	Wysolone	5mg	100.056	0.83055	0.00721	100.25±0.154

DISCUSSION

The spectrum of Prednisolone in methanol showed the absorption maxima at 244 nm. No effect of dilution was observed on the maxima, which confirmed the maxima at 244 nm. The statistical analysis of data obtained for the calibration curve of Prednisolone in pure solution indicated a high level of precision for the proposed method, as evidenced by low value of coefficient of variation. The coefficient of correlation was highly significant. The linearity range was observed between 2 – 12 mcg/ml in (fig 2). The plot clearly showed a straight line passing through origin (Y = 0.0642X + 0.0689). The estimated method was validated by low values of % RSD and standard error, indicating accuracy and precision of the methods. Excellent recovery studies further proves the accuracy of the method.

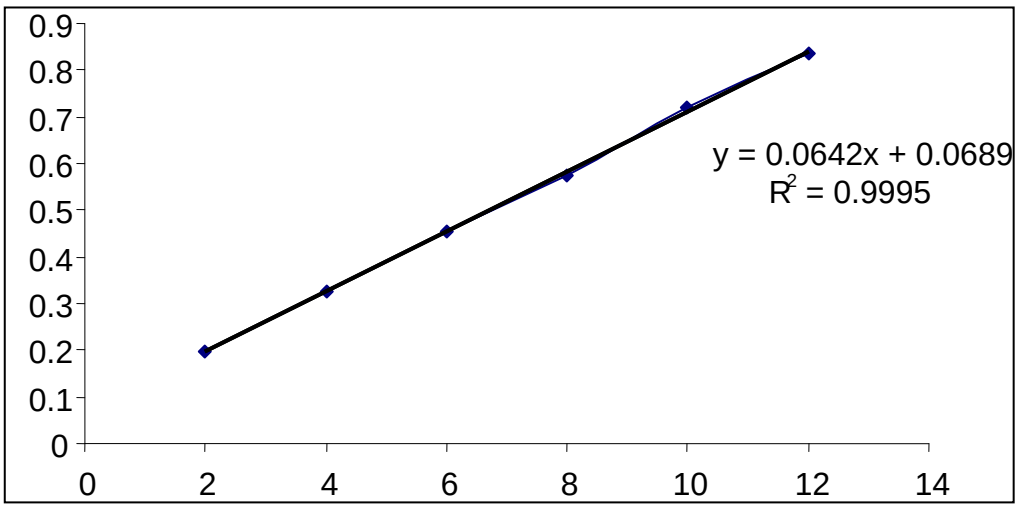

Fig. 2: Linearity curve of Prednisolone at λmax 244nm

CONCLUSION

The two proposed methods based on the Spectrophotometry were developed and validated as per ICH guidelines. The standard deviation and % RSD calculated for the proposed methods are low, indicating high degree of precision of the methods. The results of the recovery studies performed show the high degree of accuracy for the proposed methods. Hence, it can be concluded that the developed Spectrophotometric methods are accurate.

REFERENCES

1. Tripathi KD, Essentials of medical pharmacology, 6th Edition, Jaypee Brothers Medical Publishers Ltd. New Delhi, 2008; P. 217-218.
2. Sohan S. Chitlange, Kaushalendra K. Chaturvedi, Sagar B. Wankhede, Development and Validation of Spectrophotometric and HPLC method for the Simultaneous Estimation of Salbutamol sulphate and Prednisolone in Tablet Dosage Form J Chem. Pharm. Res.; 2011; 3(1) ;304-312.
3. Yoe-Ray Ku, Yi-Chu Liu And Jer-Huei Lin, Solid-phase Extraction and High-performance Liquid Chromatographic Analysis of Prednisone Adulterated in a Foreign Herbal Medicine, J of Food and Drug Anal.Vol. 9; No. 3; 2001; 150-152.
4. Mohit Rohitas, Abhinav Agrawal, Ashish K jain, Narendra K lariya, Anil K kharya and Govind P Agrawal, Development of Simultaneous Spectrophotometric Method of Mesalazine and Prednisolone in Same Dosage Form, Int. J. of App. Pharmace. Vol 2; Issue 4; 2010.
5. Moharana AK, Banerjee M, Panda S, Muduli JN, Development and Validation of UV Spectrophotometric Method for the Determination of Mesalamine in Bulk and Tablet Formulation, Int. J Pharm. Pharm. Sci, Vol 3; issue 2; 2011; 19-21.
6. Jamakhandi CM, Javali C, Disouza JI, Chougule US, Mullani AK, Spectrophotometric Determination of Lisinopril Dosage Form By Condensation Reaction, Int. J Pharm. Pharm. Sci, Vol 3; Issue 2; 2011; 185-187.
7. Eszter Desi, Agnes Kovacs, Zoltan Palotai, Aniko Kende, Analysis of Dexamethasone and Prednisolone residues in Bovine milk using Matrix Solid phase dispersion-liquid chromatography with ultraviolet detection, Micro chem. J 89 ;2008; 77–81.
8. Astha Mehta, Anil Thaker, Validated HPTLC method for assay of Prednisolone in tablets and comparison with Pharmacopeial methods J of Plan. Chromatogr. Mod. TLC Vol. 23; No. 3; Jun. 2010.