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FTIR-SPECTROPHOTOMETRIC ANALYSIS OF PREDNISOLONE IN NON-ALLOPATHIC FORMULATIONS

Pharmaceutical

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ABSTRACT

A Fourier transform infrared (FT-IR) spectrometric method was developed for the rapid, direct determination of Prednisolone in different Ayurvedic,Siddha and Naturopathic Formulations. Prednisolone is widely used in Ayurvedic preparations for the quick relief of pain in joints and arthritis. But on long term it can result in steroidal toxicity. The Universal ATR spectra of Prednisolone with a specific peak area at 1185.4-939.19 cm^{-1} were recorded and used for this study. *Quant Builder*, an OPUS software was used for data processing. An average recovery of 98.2% of Prednisolone from different dosage form with a correlation coefficient of 0.985 was obtained. The linear regression equation for Prednisolone was calculatedto be *y=-18.491-11.206*x.* The method had excellent reproducibility for the standard of 40 mg, 38.58 ±1.02 mg(*n* =6). Themethod gave rise to linear data in the range 2-50 mg with accuracy and precision in the range 0.56-2.4%. Therefore, this FT-IR-spectrophotometric assay was accurate, and may be recommended for the simple quantification of Prednisolone in Ayurvedic, Sddha and naturopathic preparations

KEYWORDS

FT-IR, Prednisolone, Calibration curve, Ayurvedic, Quant Builder

INTRODUCTION

Prednisolone is a gluco-corticoid with the general properties of the corticosteroids. It is the drug of choice for corticosteroid therapy in which routine systemic deficiency is indicated except in adrenal deficiency symptoms. By acting as a Corticosteroid Hormone Receptor Agonist it works in the body. Prednisolone is having additional anti-inflammatory and immune modulating properties. Prednisolone once bound with receptor can alter gene expression and can inhibit inflammatory response by blocking cytokine production. This agent also decreases the number of circulating white blood corpuscles[1]

Prednisolone is chemically is pregna-1,4-diene-3,11,20-trione, 17,21-dihydroxy and its molecular weight is 358.43.Prednisolone is a white to practically white, odorless, crystalline powder. It is very slightly soluble in water; slightly soluble in alcohol, in chloroform, in dioxane, and in methanol[2].

The structural formula is represented below **(Fig:1)**

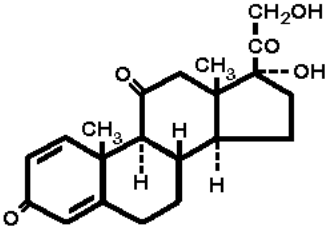


Fig:1.Chemical structure of Prednisolone

Literature survey reveals that various method for analysis of Prednisolone including HPTLC, HPLC and UV spectrophotometer. But this method proves to be more complex and is uneconomical. Here we propose an additional method for the quantification of Prednisolone in bulk as well as formulation by Fourier Transform Infrared spectrophotometer using Zinc -Selenide crystal ATR method, which is simpler and produce accurate results[3].

Infrared spectrometry (IR) provides an useful way for the identification of drugs. However, the traditional techniques employed to obtain the IR spectra, such as alkali halides disks, mulls and thin films, are sometimes not adequate for quantitative analysis[10]. Fourier Transform (FT-IR) permits continuous monitoring of the spectral baseline and simultaneous analysis of different components of the same sample[11-12].

FT-IR spectrometry provides information about components present in formulation.. Chemometric methods, such as principal component regression (PCR), Improved Principal Component Regression and partial least squares (PLS2, Multicomponent Partial Least Squares) analysis are commonly used to extract the specific information relevant to the analyte of interest from the full spectrum.Method can be validated as per ICH guidelines[4].

The main objective of this work was to develop an additional method for the fast and accurate determination of Prednisolone in commercial pharmaceutical formulations by using the Beer-Lambert law and reducing the sample pre-treatment and providing direct FT-IR measurement.

EXPERIMENTAL

Apparatus

Data acquisition was performed using an FT-IR spectrometer equipped with ATR, Zn-Se Crystal, OPUS Software (**Bruker** Co., German,). The commercial software used to generate analysis for the principal component analysis was *Quant Builder*[5].

Reagents and materials

Prednisolone pure sample was supplied by Sun Pharmaceuticals, Mumbai as gift sample and commercial non- allopathic formulations were procured from local market.

Experimental procedure

Accurately weighed 50mg of Prednisolone pure drug were kept in top of ATR crystal and spectra was recorded between 4000 and 650 cm-1, by averaging 24 scans for spectrum using OPUS software of Bruker-α /ZnSe FTIR spectrophotometer with Reflection Top-Plate . In order to compress the sample against the crystal, a pressure plate and clamp provided were used. One isolated peak **3159-3012** cm^{-1} was defined and Peak integral Area was calculated.Method Validated as per ICH guidelines[8]

Linearity and range

Different weights of Prednisolone ranging from 50 - 100 mg were taken using electronic balance with 0.001 mg sensitivity and spectrum recorded and average of such three determinations were plotted in calibration curve**(Fig:2)**.

LOD and LOQ

Limit of detection (LOD) and Limit of quantitation (LOQ) for the assay was calculated using the following formula: LOD=3.3× (standard deviation of y-intercept of the regression line / slope of the calibration curve) LOQ = 10 × (standard deviation of y-intercept of the

regression line / slope of the calibration curve)

Assay of Prednisolone content in tablet dosage form

Commercial samples of Ayurvedic, Unani and naturopathic preparations intended for rheumatic disorders were taken and twenty such tablets from each company were powdered and suitable weights were taken and spectra recorded like that of standard. Peak area integration done for the same peak and quantitative analysis performed with Quant Builder option in the software.

Accuracy

Accuracy of the developed method was carried out by performing recovery study using standard addition method, in which standard drug was added at two different concentration (50% and100%) to the pre-analyzed formulation

Precision

Precision study of the method was performed by intra-day and inter-day variation study. The intraday precision and inter-day precision was ascertained by determining AUC of 3 replicates of a fixed concentration of the drug (50mg) at three different time period of the same day and on three different days. The result of the precision studies was expressed in terms of % RSD (percentage of Relative Standard Deviation).

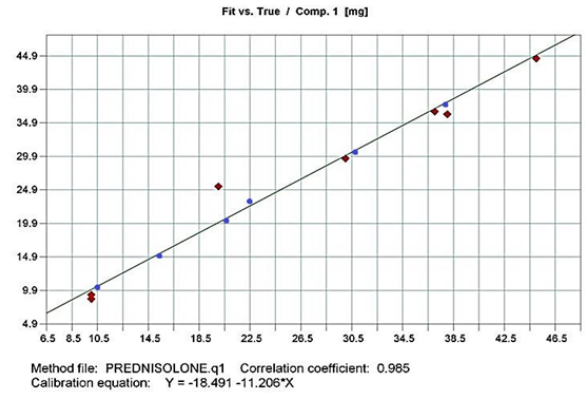
Reproducibility

Spectra were recorded for 50 mg of standard drug and it was repeated for six times.

RESULTS AND DISCUSSION

Linearity

Prednisolone was found to be linear within the concentration range 10-50 mg and exhibited correlation coefficient of 0.985 **(Fig. 2).**



LOD and LOQ

Stacked spectra of standard Prednisolone (**Fig:3**) and overlay spectra of standards(**Fig:4**) were recorded to show the precision of the method. It is of interest to note that there are no significant differences in the fingerprinting region between the spectra obtained for standard and sample, a common peak between 1185.40 and 939.19 cm^{-1} was selected for Integration**(Fig:5)**

Fig:2 Stacked spectra of prednisolone

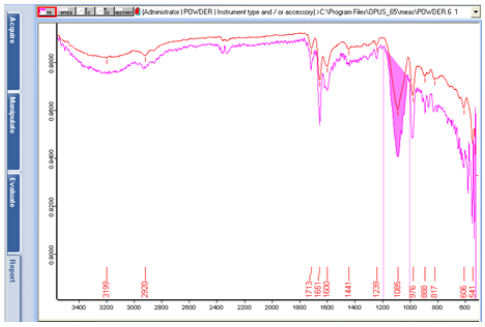
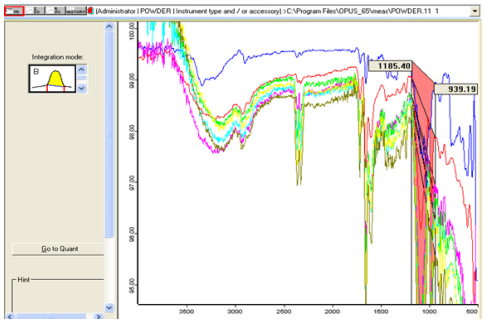


Fig:3 Overlay spectra of Prednisolone



Statistical studies like Linearity, reproducibility, correlation coefficient, student's t-test, accuracy and precision were recorded**. (Table :1)**

Recovery studies at 50 and 100% with two tablets were found out and recorded (**Table:2**).

An average of 98.8% of standard has been recovered. Amount of Drug present in the two formulations were also recorded.(**Table:3**)

Table 1:Statistical analysis

Linear regression	**y = -18.491 – 11.206 x**
Reproducibility For 40 mg	38.58 ± 1.02% (*n* = 6).
Student's t test	P = 0.05% (100.04 ± 1.29%, *n* = 3).
Accuracy and precision in the range	0.56-2.4%
R^2	0.985
Linearity	10-50 mg

Table 2: Recovery studies

Drug	Amount of sample taken(mg)	Amount of standard added(mg)	Amount Recovered (mg)	% Recovery
Formulation1	40	40	39.92	99.8
Formulation 2	40	40	39.44	98.6
Formulation 3	40	40	39.28	98.2
Formulation 4	40	40	39.76	99.4

Table 3:Estimationof Prednisolone in Ayurvedic, Siddha and Naturopathic dosage form

Formulation	Sample No	Weight of Sample taken (mg)	Amount of Prednisolone found(mg)
Ayurvedic	1	100	46.54
Ayurvedic	2	100	39.28
Siddha	1	100	38.26
Siddha	2	100	10.27
Naturopathic	1	100	10.24
Naturopathic	2	100	20.16

CONCLUSIONS

It is clear that FT-IR spectrometry is capable of direct determination of Prednisolone in formulations. With the commercial software involving chemometric approaches, Beer-Lambert law, the method proposed is simple, precise and not time-consuming compared to the chromatographic methods that exist in literature. Quantification could be done in about 5-10 minutes, including sample preparation and spectral acquisition

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