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A Novel and Validated UV - Spectrophotometric Method for Estimation of Moxifloxacin in Bulk and Tablet Dosage Form

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Abstract: A simple, precise and accurate UV Spectrophotometric method has been developed and validated for estimation of Moxifloxacin in bulk and tablet dosage form. In this method Moxifloxacin shows λmax at 290nm using 0.01N NaOH as a solvent and calibration graphs were plotted over the concentrations ranging from 1 to 10µg/ml of Moxifloxacin with correlation coefficient 0.998. The proposed method was validated as per ICH Q2 (R1) guidelines for precision, linearity, accuracy and recovery. The limit of detection (LOD) and limit of quantification (LOQ) were found to be 0.165µg/ml and 0.500µg/ml respectively by simple UV spectroscopy. The proposed method was validated.
Key words: Moxifloxacin, UV-Spectroscopy, Validation.

Introduction:

Moxifloxacin is chemically 1-cyclopropyl-7-[(S,S)-2,8-diazabicyclo[4.3.0]non-8-yl]-6-fluoro-8-methoxy-1,4-dihydro-4-oxo-3 quinoline carboxylic acid[1]. It is a slightly yellow crystalline powder with formula $C_{21}H_{24}FN_3O_4$ and molecular weight 401.43 g/mol. It has been found to be effective in acute bacterial sinusitis, acute bacterial exacerbation of chronic bronchitis, community acquired pneumonia, skin and skin structure infections. In literature survey many analytical methods includes and UV-Spectroscopic[2], RP-HPLC[3] and HPTLC[4] methods. Various methods include microbiological assays[5], voltametric determination[6] and absorption spectrometry[7] have been reported for the estimation of MOX in bulk, pharmaceutical formulation and in biological samples. In present study, simple, economical, accurate, reproducible analytical methods with better detection range for estimation of MOX in its pure form and its pharmaceutical formulations were developed. Both these developed methods were validated as per USP[8] and ICH guidelines[9].

Experimental

Materials & Methods:

The spectrophotometric measurements were carried out using a Shimadzu UV-1700 UV/Vis spectrophotometer with 1cm matched quartz cell and Shimadzu ELB 300 analytical balance, Moxifloxacin pure drug (99.89%) was obtained as a gift sample from Torrent Pharma (Baddi, India). All chemicals and reagents used were of analytical grade. Formulation used for studies was developed by Torrent Pharmaceutical Industries Ltd. Moxifloxacin tablets, Moxif (Formulation I, Torrent Pharmaceutical Industries Ltd, and Baddi) and Staxom (Formulation II, Stancare, and Delhi) were procured from local drug stores.

Preparation of Standard solution:

Standard drug of Moxifloxacin was proposed by dissolving 25mg pure Moxifloxacin in 0.01N NaOH and transferred into 250ml volumetric flask to obtain 100µg/ml of stock solution. The standard solution of Moxifloxacin having concentration of 10µg/ml was scanned in UV range (200-400nm) in 1.0 cm cell against in solvent as blank and spectrum was obtained.

Determination of λmax:

10µg/ml of Moxifloxacin was prepared and scanned in UV range of 200-400nm and spectrum was obtained. The λmax was found to be at 290nm wavelength where absorbance was found maximum at this wavelength. Hence it is considered as absorbance maxima (λmax) shown in Figure-1.

Preparation of calibration curve:

Standard stock solution was suitably diluted with 0.01N NaOH to obtain concentrations ranging from 1-10µg/ml. Absorbance of these solutions was measured at 290nm. Calibration curve was obtained by plotting graph between concentration and absorbance shown in Figure-2.

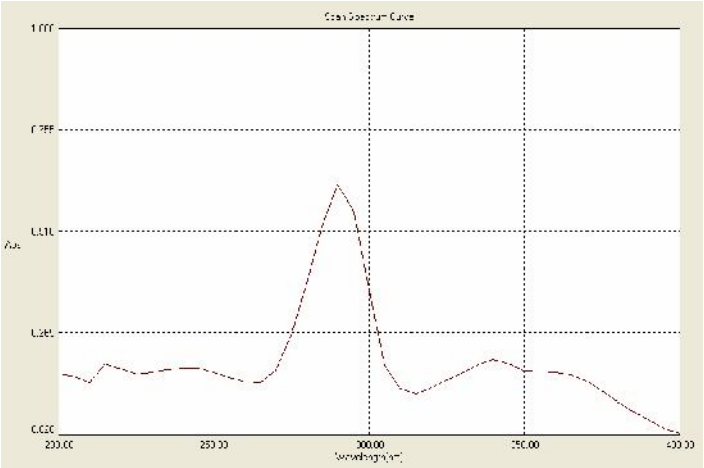

Fig - 1: UV Spectrum of Moxifloxacin in 0.01N NaOH

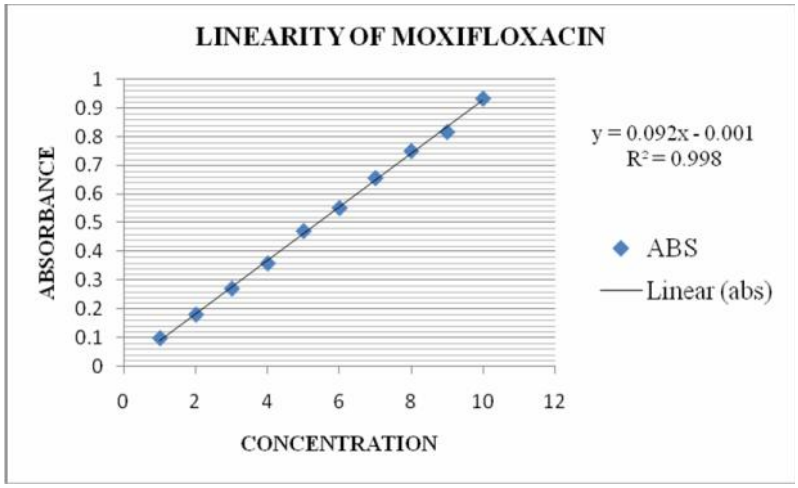

Fig - 2: Calibration curve of Moxifloxacin in 0.01N NaOH showing linearity relationship

Preparation of test solution:

20 Tablets were weighed and its average weight was determined. An accurately weighed tablet powder equivalent to 25mg of Moxifloxacin transferred into 250ml volumetric flask dissolved in 0.01N NaOH, sonicated for 10min and volume was made up to the mark. Solution was filtered using whattman filter paper (No.41) to obtain 100µg/ml stock solution.

Validation:

Linearity:

The absorbances were observed from 1 to 10µg/ml and were shown in Table-1. Linearity was obtained between 1 to 10µg/ml. Concentration graph was plotted for concentration and absorbance. The equation of calibration curve obtained was y = 0.092x + 0.001. The correlation coefficient (r) was 0.998 shown in Figure-2.

Accuracy:

To determine the accuracy of the method recovery was performed by standard addition method. To pre-analyzed sample known amount of standard Moxifloxacin was spiked in different concentrations. The recovery was performed at three levels 50%, 100% and 150% of standard Moxifloxacin. Solutions were analyzed and percentage recovery was calculated from calibration curve shown in Table-2.

Precision: It was ascertained by replicate analysis of the homogenous sample of tablet powder and the concurrent values of estimation are shown in Table-3 for two different brands of the sample by proposed method.

Interday & Intraday Precision

The concentration of 10µg/ml of Moxifloxacin (on label claim basis) was taken. The absorbance of the final solution was read after 0hr, 12hr and 24hr in 1.0 cm cell at selected wavelength. The results were recorded in Table-5.

Similarly the absorbance of the same solution was read on 1st, 2nd and 3rd day. The amount of Moxifloxacin was estimated by comparison with the standard and taking A(1%, 1cm) at 290nm. The results were recorded in Table-6.

Ruggedness:

It was carried out by analyzing the sample by three different days and estimation of drug by proposed methods. Results of studies are shown in Table-7.

Table 1: Calibration data for analysis of Moxifloxacin in 0.01N NaOH at λmax = 290nm

Concentration (µg/ml)	Mean Absorbance (± SD)
1	0.099 (0.0006)
2	0.181 (0.0021)
3	0.272 (0.001)
4	0.358 (0.0015)
5	0.473 (0.0017)
6	0.552 (0.001)
7	0.656 (0.0015)
8	0.749 (0.0006)
9	0.816 (0.002)
10	0.932 (0.001)

Table-2: Recovery data of Moxifloxacin in 0.01N NaOH

Ingredient	Amount of drug from formulation	Amount of standard added	Percentage added	Amount added	Amount found	% Recovery (Mean ± RSD)*
Moxifloxacin	4µg	2µg	50%	1.99	2.00	99.93 ± 0.35
Moxifloxacin	4µg	4µg	100%	3.99	3.99	100.02 ± 0.05
Moxifloxacin	4µg	6µg	150%	5.99	5.99	100.11 ± 0.37

*n=3 (Average of 3 determinations)

Table-3: Results of analysis of laboratory samples (Assay)

Sample	Label	Amount found	% Label claim
Brand-1 Moxifloxacin	400mg	399.71	99.82 ± 0.16
Brand-2 Moxifloxacin	400mg	399.85	99.85 ± 0.35

*n=3 (Average of 3 determinations)

Table-4: Lowest Limit of detection and Lowest Limit of quantification

LOD (µg/ml)	LOQ (µg/ml)
0.165	0.500

Table-5: Results of Inter-day Precision of Moxifloxacin in 0.01N NaOH

Parameter	% Recovery Estimated (Mean + RSD)*		
	6 (µg/ml)	8 (µg/ml)	10 (µg/ml)
At 0 hr	99.33 ± 0.44	99.83 ± 0.33	99.96 ± 0.53
At 12 hr	99.96 ± 0.38	99.70 ± 0.50	99.89 ± 0.50
At 24 hr	99.63 ± 0.51	99.79 ± 0.59	100.03 ± 0.23

*n=3 (Average of 3 determinations)

Table-6: Results of Intraday Precision of Moxifloxacin in 0.01N NaOH

Parameter	% Recovery Estimated (Mean + RSD)*		
	6 (µg/ml)	8 (µg/ml)	10 (µg/ml)
Day-1	99.48 ± 0.15	99.52 ± 0.49	99.71 ± 0.51
Day-2	99.42 ± 0.37	99.66 ± 0.19	99.60 ± 0.53
Day-3	99.33 ± 0.51	99.78 ± 0.40	99.85 ± 0.23

*n=3 (Average of 3 determinations)

Table-7: Results of Ruggedness of Moxifloxacin in 0.01N NaOH

Ruggedness	% RSD*
Analyst – 1	0.36
Analyst – 2	0.24

*n=3 (Average of 3 determinations)

Table-8: Validation Parameters

Parameters	Results
Beer's law limit (µg/ml)	1-10
Absorptivity ($1mole^{-1}$, cms^{-1})	0.932×10^4
Sandell's sensitivity (µg/cm2/0.001)	0.0107
Correlation coefficient	0.998
Regression equation	Y = 0.092x - 0.001
Limit of detection (µg/ml)	0.165
Limit of quantification (µg/ml)	0.500
Precision (% RSD)	0.001

Results and Discussions:

Attempt has been made to develop rapid, sensitive, economic, precise and accurate analytical method for Moxifloxacin in pure and pharmaceutical dosage form. The proposed method is based on UV Spectrophotometric absorption in UV region using 0.01N NaOH as solvent. Maximum absorbance was found to be at 290nm. LOD and LOQ were found to be 0.165µg/ml and 0.500µg/ml. Beer's law was obeyed in concentrations ranging from 1 to 10µg/ml. The correlation coefficient values were above 0.998 which shows that absorbance was linear with concentration. The optical characteristics such as Beer's law limit, correlation coefficient, slope, intercept, molar absorptivity, scandell's sensitivity were calculated and validated (**Table-8).** Precision of the method was confirmed by Intraday and Interday analysis, %RSD values were found to be less than 2.0. The percent recovery was found to be nearly 100% indicating reproducibility and accuracy of the methods. Hence the proposed method could be effectively adopted for routine quality control of Moxifloxacin in bulk and formulated tablet dosage form.

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References:

1. S. Budavari, Eds, In; The Merck Index.13th edition, Merck and co., Inc., White house station, NJ , 2001, 1097 and 1125.
2. P. U. Patel, et al: Simultaneous spectrophotometric determination of Moxifloxacin and Metronidazole in synthetic mixture by simultaneous equations method. Indian drugs, 2005, 42(3), 155-157.
3. M.N. Saraf, et al: Determination of Moxifloxacin in plasma by RP-HPLC with fluorescence detection for Bioequivalence studies in healthy Human subjects, Indian drugs, 2005, 42(6), 375-379.
4. M.V. Baldaniya, et al: HPTLC method for estimation of Moxifloxacin in tablet dosage form, Indian J. Pharma. Sciences, 2005, 67(1), 112-115.
5. Guerra F, Paim CS, Steppe M, Schapoval EE. Biological assay and liquid chromatographic method for analysis of moxifloxacin in tablets. J. AOAC. Int. 2005, 88, 1086-92.
6. Erk N.: Voltammetric behaviour and determination of Moxifloxacin in pharmaceutical products and human plasma. Anal. Bioanal. Chem. 2004, 378, 1351-56.
7. Al-Ghannam SM. Atomic absorption spectroscopic, conductometric and colorimetric methods for determination of some fluoroquinolone antibacterials using ammonium reineckate, J. Chromatog. B. Analyt. Technol. Biomed. Life. Sci. 2008, 69(04), 1188-94.
8. ICH Q2R1 2005 Validation of Analytical Procedures Text and Methodology, in: International Conference on Harmonization of Technical Requirements for Registration of Pharmaceuticals for Human Use.
9. United States Pharmacopoeia, Validation of Compendial Methods (Pharmacopoeial Convention Inc., Rockville, MD) 2004, 2622-2625.

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