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ESTIMATION OF LORNOXICAM AND DIACEREIN IN BULK AND PHARMACEUTICAL DOSAGE FROM BY SIMULTANEOUS EQUATION AND Q-ANALYSIS USING UV SPECTROSCOPIC TECHNIQUE

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ABSTRACT

We have developed two simple accurate and economic UV spectrophotometric methods for the simultaneous estimation of Lornoxicam and Diacerein in bulk and pharmaceutical formulation. The solvent used is methanol and the λ max or the absorption maxima of the Lornoxicam and Diacerein was found to be 382 nm and 341 nm respectively. Two wavelengths were selected at wavelengths 341 nm and isobestic point 274 nm, in absorbance ratio method. The Beer- Lambert's law followed in the concentration range of 2-10 µg/ml and 10-50 µg/ml for Lornoxicam and Diacerein respectively. These two methods can be used for the analysis of both drugs in pharmaceutical dosage form and quality control study.

KEYWORDS

Lornoxicam, Diacerein, UV spectrophotometer, Simultaneous equation method and Q-analysis method.

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INTRODUCTION

Lornoxicam is used as non-steroidal anti-inflammatory and analgesic[1]. Lornoxicam is used in the treatment of various types of pain, especially resulting from inflammatory diseases of the joints, osteoarthritis, surgery, sciatica, and other inflammations. Chemically, Lornoxicam is (3E)-6-chloro-3-[hydroxy(pyridin-2-ylamino)methylene]-2-methyl-2,3-dihydro-4Hthieno[2, 3 e] [1, 2]thiazin-4-one 1, 1-dioxide[1] as shown in Figure No.1.

Diacerein is an anti-inflammatory agent. It works by blocking the actions of interleukin-1 beta, a

protein involved in the inflammation and destruction of cartilage that play a role in the development of symptoms of degenerative joint diseases such as osteoarthritis[2]. Chemically, Diacerein is 4, 5- diacetoxy-9, 10-dihydro9, 10 di-oxo-2 anthracene carboxylic acid as shown in Figure No.2.

Literature survey reveals that some methods have been developed for the determination of Diacerein and Lornoxicam by UV Spectrophotometry[3-8] or HPLC[9-13] alone.

The objective of work is to develop simple accurate and precise UV spectrophotometric methods for simultaneous estimation of Lornoxicam and Diacerine in Pharmaceutical formulations.

MATERIAL AND METHODS

Standard drug Lornoxicam and Diacerine were gifted from Emcure Pharmaceutical, Pune. Other chemicals used were analytical or HPLC used were Class A grade. Shimadzu UV - 1700 spectrophotometer with UV probe 2.10 software and 1 cm matched quartz cells were used for absorbance measurements. Analytical balance used for weighing standard and sample.

Preparation of standard stock solution

Accurately about 10 mg of reference standard Lornoxicam and Diacerein were weighed and transferred to 100 ml volumetric flask separately and dissolved in 20 ml methanol which further sonicated for 20 min. The volume was made up to 100 ml with methanol to give stock solution of concentration 100 µg/ml Lornoxicam and Diacerein respectively.

Selection of analytical wavelength

Each solution of 10 µg/ml Lornoxicam and 10 µg/ml Diacerein were scanned in spectrum mode of UV visible spectrophotometer instrument from 400 nm to 200 nm. The maximum absorbance of Lornoxicam and diacerein was observed at 382 nm and 341 nm respectively as shown in Figure No.3 and Figure No.4 respectively. The overlain spectra of both the drug is given in Figure No.5.

Method of estimation and validation

Method A: Simultaneous equation method

In simultaneous equation method, two wavelengths were selected 382 nm add 341 nm for Lornoxicam and Diacerein respectively. A series of standard solution containing 2-10 µg/ml of Lornoxicam and 10-50 µg/ml of Diacerein respectively. The content of ingredient was calculated by substituting values in the following equation:

$$C_x = \frac{A2ay1 - A1ay2}{ax2ay1 - ax1ay2}$$

$$C_y = \frac{A1ax2 - A2ax1}{ax2ay1 - ax1ay2}$$

Where,

A_1= absorbance of sample at 341 nm

A_2= absorbance of sample at 382 nm

ax_1= absorptivity of diacerein at 341 nm

ay_2= absorptivity of Lornoxicam at 382 nm

Cx=concentration of diacerein in the diluted sample

Cy= concentration of Lornoxicam in the diluted sample

Method B: Absorbance ratio method (Q-analysis method)

In absorbance ratio method absorbance of Lornoxicam and Diacerein were measured at λ Max of Diacerine 341 nm and Isobestic point 274 nm respectively using methanol as blank. The concentrations of each drug were calculated by substituting values in the equation below:

$$C_x = \frac{Qm - Qx}{Qy - Qx} \times \frac{A1}{ax1}$$

$$C_y = \frac{Qm - Qy}{Qx - Qy} \times \frac{A1}{ay1}$$

Where,

Qm= Ratio of absorptivity of mixture at 341nm and 274 nm

Qx= Ratio of absorptivity Diacerein at 341nm and 274 nm

Qy= Ratio of absorptivity of Lornoxicam 341nm and 274nm

Assay of marketed formulation

Twenty tablets (each tablet containing 40 mg Lornoxicam and 100 mg of Diacerein) in combined dosage form were weighed and average weight was determined. Tablets were crushed into fine powder in glass mortar. The tablet powder equivalent to 4 mg of Lornoxicam and 10 mg of Diacerein was weighed and dissolved in 50 ml methanol. This was

further sonicated for 30 min and then filtered through whatman filter paper No. 41. The volume was made up to 100 ml with methanol. Further dilution was made to give a concentration of 4 µg/ml Lornoxicam and 10 µg/ml of Diacerein and the analysis procedure was repeated six times for Tablet formulation.

METHOD VALIDATION

Specificity

The specificity of method was performed by comparing the chromatograms of blank, standard and sample. There was no any interference from excipients.

Linearity

The linearity of Lornoxicam was studied in the concentration range of 2-12 µg/ml at 382 nm and linearity of Diacerein was studied in the concentration range of 10-60 µg/ml at 341 nm.

Accuracy

The accuracy for the analytical procedure was determined at 50%, 100% and 150% of the label claim. Results were expressed in terms of % recoveries. Three determinations at each level were performed and % RSD was calculated.

LOD and LOQ

In this study, LOD and LOQ were based on the standard deviation of the response and the slope of the corresponding curve using the following formula:

$LOD = 3.3 \times SD/S$

$LOQ = 10 \times SD/S$

Where,

SD = standard deviation of y-intercept of the calibration curves.

S = mean slope of six calibration curves calibrations graphs.

RESULTS AND DISCUSSION

Simultaneous equation method and Q-analysis method were developed for estimation of Lornoxicam and Diacerein in bulk and pharmaceutical formulations. The developed UV-spectroscopic methods were validated for parameter like linearity, specificity, range, accuracy etc. The method validated according to ICH Q2B guidelines for validation of analytical procedure[14-16]

Specificity of the method was established by evaluating the interference of the placebo. No interference of placebo was detected. The linearity was found to be 2-12 µg/ml for Lornoxicam and 10-60 µg/ml for Diacerein respectively. Correlation coefficients for the regression line were found to be 0.9990 and 0.9993 for Lornoxicam and Diacerein respectively which shown in Figure No.6 and Figure No.7 respectively and data for calibration curve tabulated in Table No.1.

The recovery was determined at three levels, viz. 50%, 100%, and 150% of the label claim. Three samples were prepared for each recovery level. The recoveries study by simultaneous equation method and Q-analysis method were found to be between 99.0 % to 103 %.

The results of Method A and Method B were tabulated in Table No.3 and Table No.4 respectively. Six replicates at assay concentration were carried out. The results of assay are presented in Table No.5 for method A and Table No.6 for method B. In method A, Detection limit for Lornoxicam was 0.062 µg/ml; Quantification limit was 0.19 µg/ml and in method B Detection limit for Diacerein was 0.20 µg/ml Quantification limit was 0.608 µg/ml.

The result of analysis show the proposed analytical methods are capable of estimating Lornoxicam and Diacerein in bulk and finished dosage form.

Table No.1: Data for Calibration Curve

S.No	Lornoxicam		S.No	Diacerein	
	Concentration µg/ml	Absorbance		Concentration µg/ml	Absorbance
1	2	0.107	1	10	0.182
2	4	0.221	2	20	0.329
3	6	0.338	2	30	0.498
4	8	0.414	4	40	0.654
5	10	0.527	5	50	0.819
6	12	0.611	6	60	0.984

Table No.2: Optical characteristic and other parameter

S.No	Parameters	Lornoxicam	Diacerein
1	λ Max (nm)	382	342
2	Range (µg/ml)	2-12	10-60
3	Correlation Coefficient (r^2)	0.9990	0.9993
4	Intercept(c)	0.008	0.014
5	Slope	0.051	0.016
6	LOD(µg/ml)	0.062	0.20
7	LOQ(µg/ml)	0.19	0.608

Table No.3: Recovery study data Method A

	Lornoxicam				Diacerein			
S.No	Level (%)	Amount of std drug added mg	% Recovery	% RSD	Level (%)	Amount of std drug added mg	% Recovery	% RSD
1	50	2	99.86	0.981	50	5	100.2	0.758
2	100	4	100.2	0.881	100	10	101.2	1.642
3	150	6	99.84	1.201	150	15	99.37	0.841

* Mean of six estimations (n=3) for each level.

Table No.4: Recovery study data Method B

	Lornoxicam				Diacerein			
S.No	Level (%)	Amount of std drug added mg	% Recovery	% RSD	Level (%)	Amount of std drug added mg	% Recovery	% RSD
1	50	2	99.24	0.489	50	5	99.73	0.638
2	100	4	98.94	0.592	100	10	100.06	0.539
3	150	6	98.96	0.470	150	15	99.56	0.566

* Mean of six estimations (n=3) for each level.

Table No.5: Assay study data Method A

Lornoxicam	Diacerein		Lornoxicam		Diacerein	
S.No	Amount Present (µg/ml)		Amount Found (µg/ml)	% Purity	Amount Found (µg/ml)	% Purity
1	4	10	4.043	101.07	9.826	98.26
2	4	10	4.128	103.20	9.865	98.65
3	4	10	4.159	103.97	10.103	101.03
4	4	10	4.081	102.02	10.157	101.15

5	4	10	4.157	103.92	9.921	99.21
6	4	10	4.194	104.85	9.878	98.78
7	Mean		4.123	103.17	9.958	99.19
8	SD		0.05049	1.26238	0.1472	0.8497

Table No.6: Assay study data Method B

Lornoxicam	**Diacerein**	**Lornoxicam**		**Diacerein**	
S.No	**Amount Present (µg/ml)**	**Amount Found (µg/ml)**	**% Purity**	**Amount Found (µg/ml)**	**% Purity**
1	4 10	4.192	104.80	10.09	100.98
2	4 10	4.159	103.97	10.01	100.10
3	4 10	4.037	100.92	9.92	99.23
4	4 10	4.132	103.30	9.76	97.60
5	4 10	4.084	102.10	9.82	98.20
6	4 10	4.185	104.62	10.16	101.67
7	Mean	4.131	102.28	9.96	99.63
8	SD	0.0627	1.53334	1.1583	1.5734

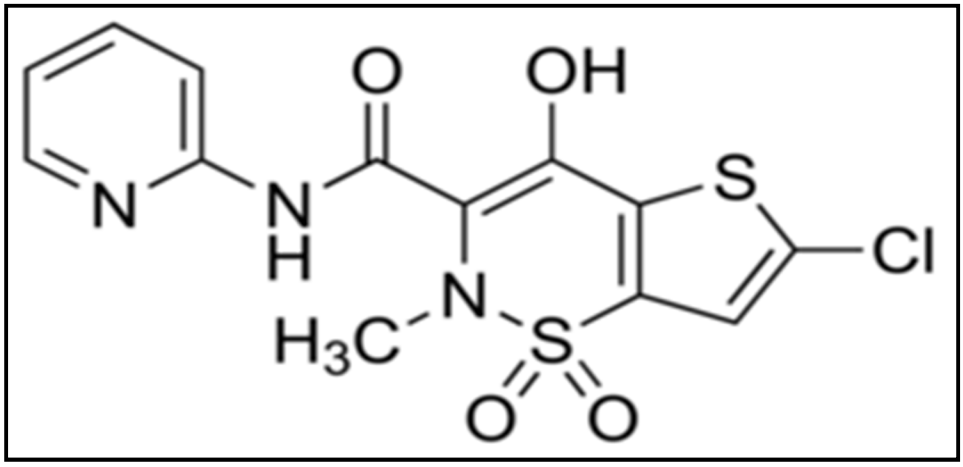

Figure No.1: Chemical structure of Lornoxicam

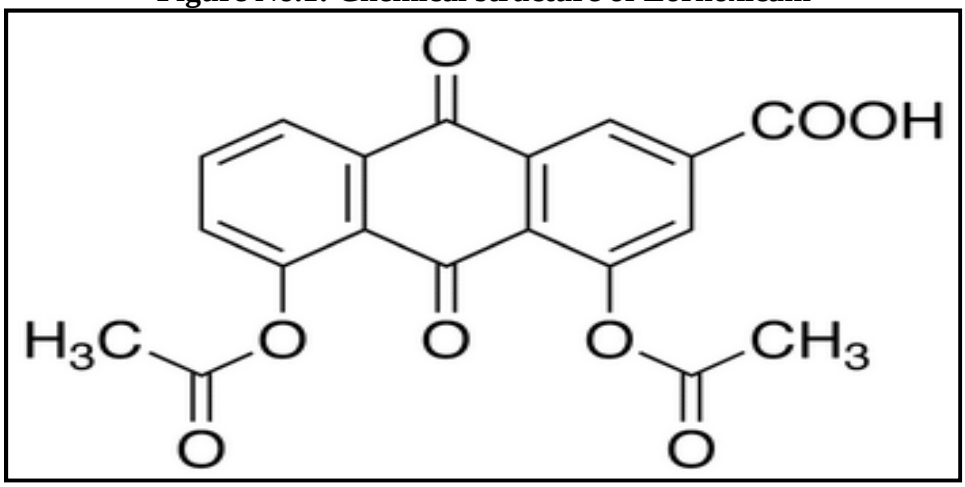

Figure No.2: Chemical structure of Diacerein

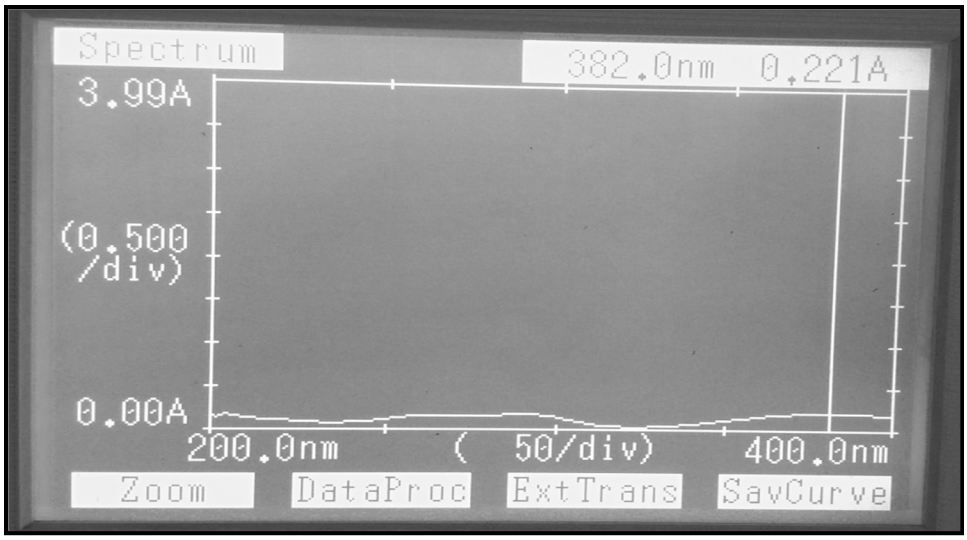

Figure No.3: Absorption maxima of Lornoxicam in methanol

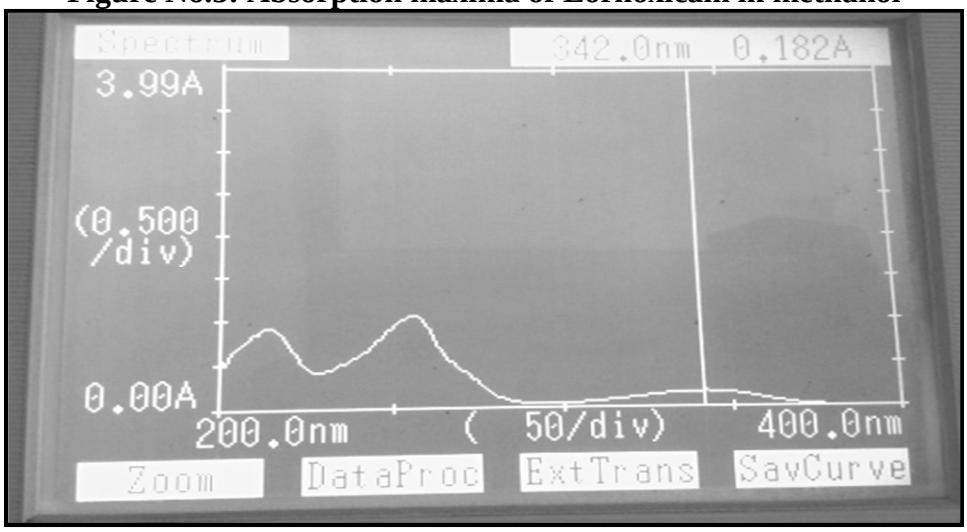

Figure No.4: Absorption maxima of Diacerein in methanol

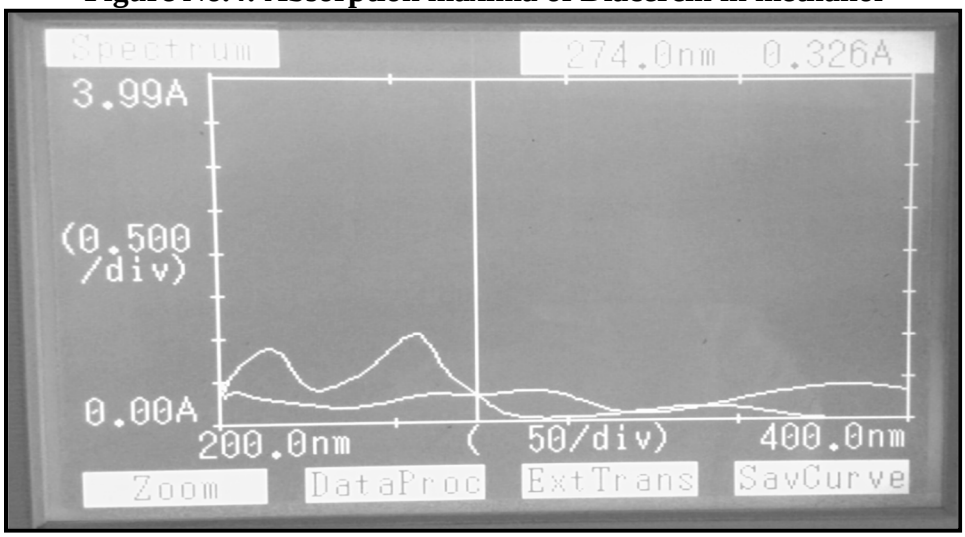

Figure No.5: Overlain spectra of Lornoxicam and Diacerein

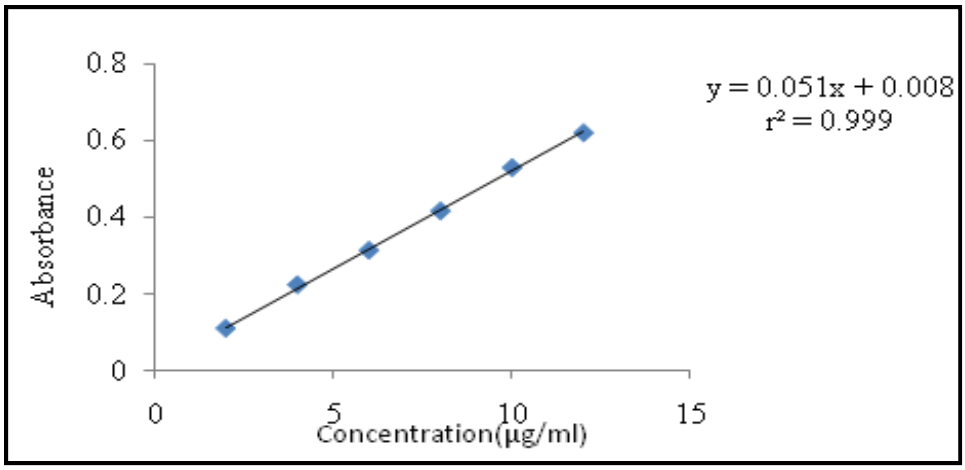

Figure No.6: Calibration curve of Lornoxicam

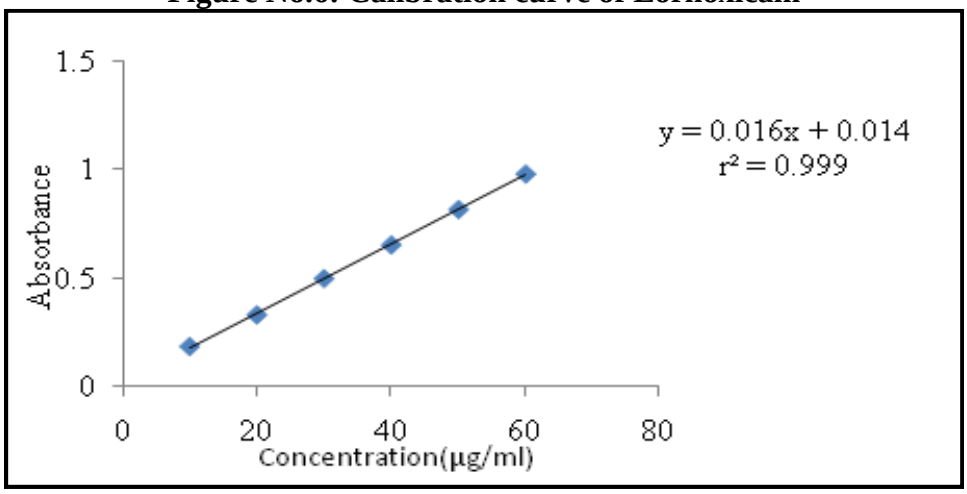

Figure No.7: Calibration curve of Diacerein

CONCLUSION

The UV spectrophotometric methods i.e simultaneous equation method and Q-analysis methods are simple accurate, precise and economical. It showed good accuracy close to 100% for both Lornoxicam and Diacerein. This developed method can be used for simultaneous analysis of Lornoxicam and Diacerein in bulk and pharmaceutical dosage form.

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CONFLICT OF INTEREST

We declare that we have no conflict of interest.

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