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ORIGINAL ARTICLE

UV Visible Spectrophotometry Method Development and Validation of Felodipine in Bulk Drug

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ABSTRACT
The objective of this study was to establish a validated method development for routine analysis of Felodipine in bulk drug samples. A simple, accurate, precise, and cost effective UV-visible spectrophotometric method was the estimation of Felodipine in its pure.The physicochemical characterization study of Felodipine was performed in UV-visible spectrophotometry, FT-IR and melting point techniques. The absorption maxima of the drug are found to be 362.20 nm in the solvent methanol. The concentration range 5-25 µg/ml was produced linear response with regression coefficient was 0.9965.Method validation parameters were performed as per the guideline of International Conference for Harmonization. The melting point was found to be 143^0 C in the standard range. The IR spectrum of Felodipine with potassium bromide was found the sharp and accurate peaks.
Key words: - Felodipine, λmax, IR spectra, UV-visible spectrophotometer

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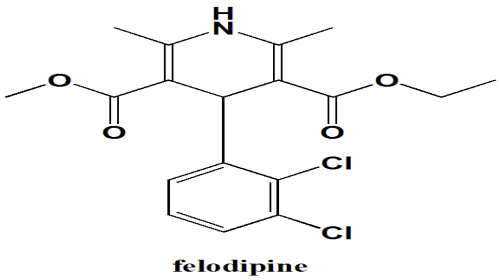
INTRODUCTION

UV Spectrophotometric in that method validation performed which is helps to validation of analytical method for a range of concentrations and that is the change in formulation or concentration do not require additional validation. Also analytical method development needed to understand the critical process parameters and which is minimize their influence on accuracy and precision.



felodipine

Fig. 1: Chemical structure of Felodipine

The IUPAC name of Felodipine is [(RS)-3-ethyl 5-methyl 4-(2, 3-dichlorophenyl)-2, 6-dimethyl-1, 4-dihydropyridine-3, 5-dicarboxylate]. Felodipine having molecular formula $C_{18}H_{19}Cl_2NO_4$and their molecular weight is 284.3 g/mol [1]. The melting point of Felodipine is 141^0C-145^0C [8]. Felodipine is soluble in methanol. Felodipine is in class of calcium channel blocker. [10, 11, 12]

The Felodipine is used to treat myocardial infraction disease, stable angina, high blood pressure and heart failure [7]. Felodipine inhibits calcium ions from entering the slow channels of vascular smooth muscle and myocardium during depolarization. Felodipine is producing a relaxation of coronary vascular smooth muscle and coronary vasodilation [13].

Estimation of Felodipine by reported, however there was robust, simple, accurate, precise, and cost effective for Felodipine in bulk drug by UV spectrometer for analytical method development and validation. For the routine analysis, this method was very simple.

MATERIAL AND METHODS

Experimental

Instrument and Materials

The UV spectrophotometer (SHIMADZU UV 1800) is double beam spectrometer which is standard and reference solutions used. 1 cm quartz cells are used. UV probe software are using in PC which is controlled by UV-Visible Spectrophotometer. FT-IR (PERKIN ELEMER SPECTRUM 65) is recorded IR spectra by using KBr press pellet technique.Melting point apparatus(VEEGO) VMP-D/S is use to checking physical constant.

Felodipine and methanol purchased from Vishal Institute of Pharmaceutical Education And Research, Ale

Solution preparation

Preparation of Stock Solution

0.25 mg Felodipine is weighed accurately and transferred into 25 ml volumetric flask. Then about 20 ml methanol is adding and shakes well. After that volume make up by using same solvent. This stock solution concentration is make 1000 µg/ml.

Preparation of Sample Solution

From above stock solutionwithdraw 10 mland transferred into 100 ml volumetric flask then volume make up by solvent and shake well. Then the concentrations of this solution are becomes 100 µg/ml. Then again withdraw 1.25 ml, 2.5 ml, 3.75 ml, 5 ml, and 6.25 ml from 100 µg/ml solution and transfer into 25 ml volumetric flask separately after that volume make up by same solvent and shakes well. Then this solution concentration becomes 5µg/ml, 10µg/ml, 15µg/ml, 20µg/ml and 25µg/ml respectively.

Selection of Wavelength[2, 5, 6]

Take 6.25 ml from 100 µg/ml solution and transfer into 25 ml volumetric flask and volume make up by using solvent and shake well. Then this 25µg/ml solution are scanned in the range 200-400 nm on spectrum mode by using methanol as a reference solution. Then after scanned the result λmax was 362.20 nm. This wavelength was selected for analytical study of method validation parameter.

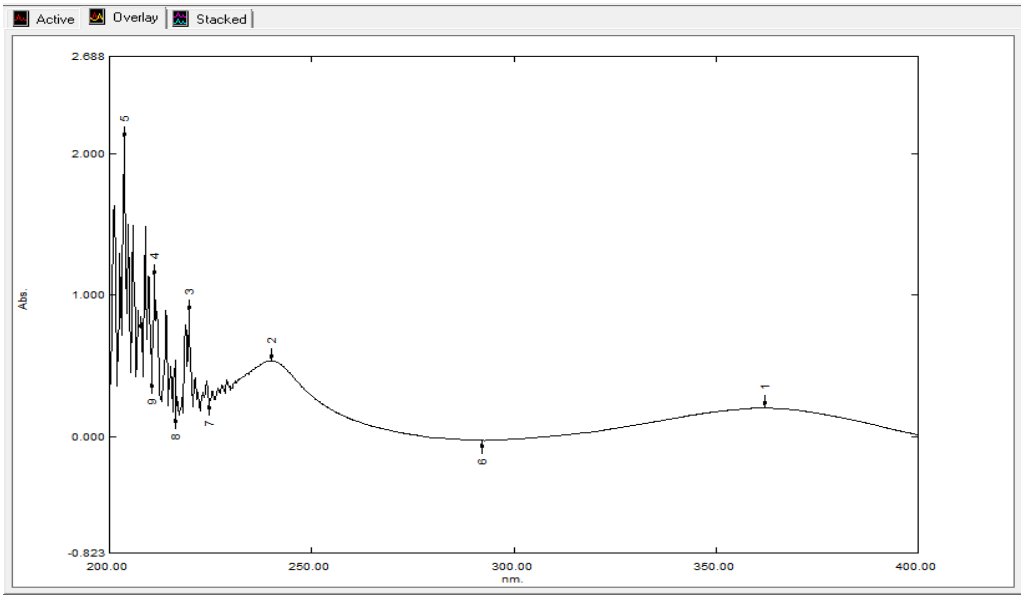


Fig. 2: UV Spectrum of Felodipine

RESULTS

Linearity

Linearity is an analytical method which is gives results that are directly proportional to the concentration of analyte in the solution. Linearity was performed by using sample solution. The solution 5 µg/ml to 25 µg/ml concentration range was prepared. Then absorbance is showing proportionally to the concentration range.

Table no. 1: Spectrometric data for linearity of Felodipine at 362.20 nm

Sr. No.	Concentration (µg/ml)	Absorbance
1	Blank	0.00
2	5	0.043
3	10	0.113
4	15	0.180
5	20	0.228
6	25	0.296

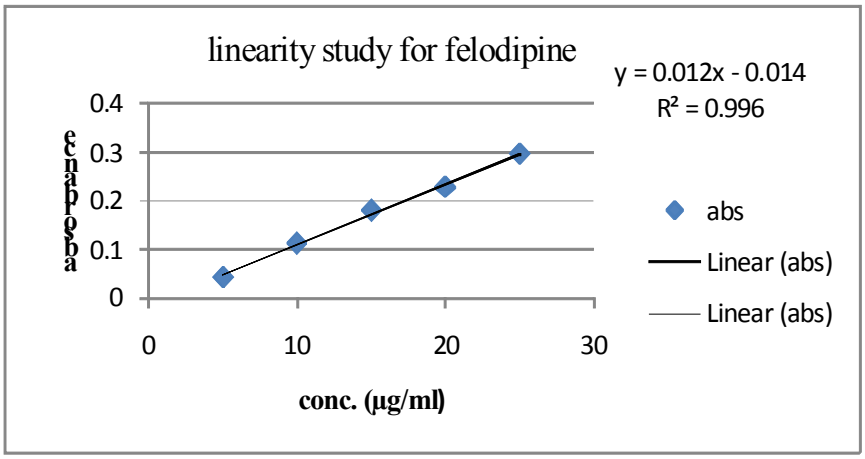


Fig . 3 Calibration curve of Felodipine

Table. 2: Linearity of Felodipine

Sr. No.	Concentration (µg/ml)	Absorbance
1	5	0.043
2	10	0.113
3	15	0.180
4	20	0.228
5	25	0.296
Regression Data	M	0.0124
	C	-0.0143
	R^2	0.9965

Precision

Precision is the analytical method in which the procedure is applied repeatedly to multiple samplings from a homogeneous lot. We were take three concentration and triplicate absorbance of same concentration range are used in intra-day and inter-day precision.

Table . 3 Intra-day Precision

Sr. No.	Concentration (µg/ml)	Absorbance	Average	SD	RSD	%RSD
1	15 15 15	0.193 0.196 0.186	0.191666	0.005131	0.026773	2.677357
2	20 20 20	0.252 0.260 0.273	0.261667	0.010599	0.040505	4.050475
3	25 25 25	0.333 0.343 0.342	0.339333	0.005508	0.016231	1.623056

Table no. 4 Inter-day Precision

Sr. No.	Concentration (µg/ml)	Absorbance	Average	SD	RSD	%RSD
1	15 15 15	0.198 0.197 0.199	0.198	0.001	0.005050	0.505050
2	20 20 20	0.278 0.292 0.298	0.289333	0.010263	0.035472	3.54719
3	25 25 25	0.355 0.362 0.349	0.355333	0.006506	0.018311	1.831071

Robustness

Robustness is the method to remains which unaffected by small, deliberate variations in method parameters. In that, the measure is of the reliability of a method.

Table no. 5 Robustness study of Felodipine

Sr. No.	Concentration (µg/ml)	361 nm	362.20 nm	363.5 nm
1	10	0.115	0.127	0.129
2	10	0.117	0.118	0.132
3	10	0.114	0.118	0.143
4	10	0.112	0.136	0.137
5	10	0.119	0.148	0.152
Average		0.1154	0.1294	0.1386
SD		0.0027018	0.012798	0.009182
RSD		0.023412922	0.098906	0.066245
%RSD		2.341292216	9.890601	6.624461

Ruggedness

Ruggedness are performed because the cross checking of constancy of the results when external factors such as analyst, laboratory, instrument, reagent and days are varied. We were taking the different analyst which taken a reading at same concentration and calculated % RSD.

Table No. 6 Ruggedness study of Felodipine

Sr. No.	Concentration (µg/ml)	Analyst 1	Analyst 2	Analyst 3
1	5	0.049	0.048	0.049
2	5	0.051	0.050	0.052
3	5	0.065	0.061	0.064
Average		0.055	0.053	0.055
SD		0.0087177	0.007	0.007937
RSD		0.1585054	0.132075	0.144314
%RSD		15.850541	13.20755	14.43137

Limit of Detection [3]

LOD is the lowest concentration at which the instrument is able to detect but not quantify.LOD is calculated manually by using calibration of linearity of the Felodipine graph.

LOD=3.3 × SD/ SLOPE

Table . 7 LOD of Felodipine

Sr. No.	Concentration (µg/ml)	Absorbance
1	5	0.043
2	10	0.113
3	15	0.180
4	20	0.228
5	25	0.296
SD		0.09835
Slope		0.0124
LOD		26.17

Limit of Quantitation [3]

LOQ is the lowest concentration at which the instrument is able to detect and quantify. LOQ is calculated manually by using calibration of linearity of the Felodipine graph.

LOQ=10 × SD / SLOPE

Table No. 8 LOQ of Felodipine

Sr. No.	Concentration (µg/ml)	Absorbance
1	5	0.043
2	10	0.113
3	15	0.180
4	20	0.228
5	25	0.296
SD		0.09835
Slope		0.0124
LOQ		79.90

FT-IR[4, 9]

FT-IR spectra of Felodipine were recorded in KBr medium pellets by using FT-IR spectrophotometer. The major functional groups like primary amine, chloro and carbonyl groups are obtained in given IR spectra.

Table No. 9 IR observation of Felodipine

υ(cm^{-1}) Experimental	υ(cm-1) Theoretical	Functional group
3371.36	3374	Free N-H stretching
3069.18	3111	Aromatic C-H stretching
2947.92, 2979.75	2959, 2872	Aliphatic C-H stretching
1853.54	1714	Ester stretching
1381.42	1382	C-CH_3 stretching
1496.44	1489.10	C=C

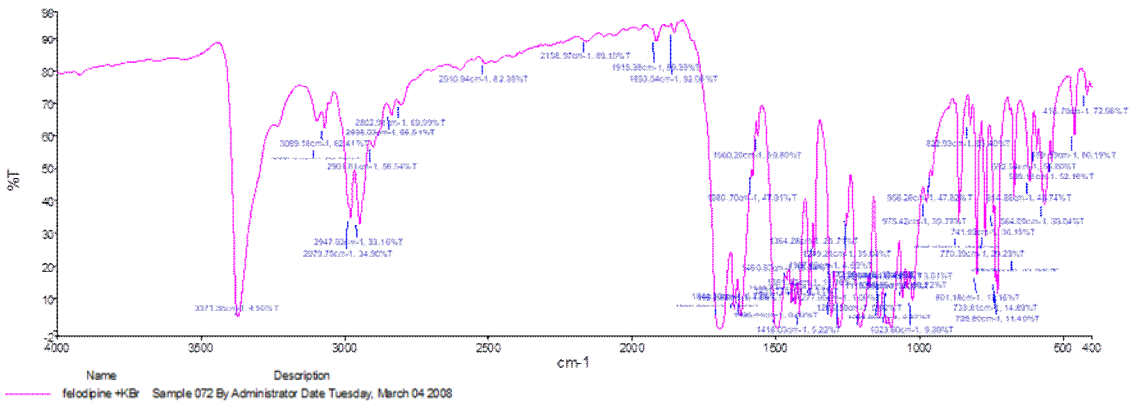


Fig no. 4 IR Spectra of Felodipine

Melting Point[8]

Capillary tube is filling by Felodipine and melting point taken by using melting point apparatus. The melting point of Felodipine was 143^0 C recorded.

DISCUSSION

It was our enthusiastic objective to establish a precise and sensitive method for routine analysis of Felodipine. UV-visible spectrophotometric method for the estimation of Felodipine in its pure was simple, accurate, and cost effective. This method validation was followed by ICH guidelines. The validation data of Felodipine was given in table 1-9 and fig 2-4 which shows the detection wavelength maxima at 362.20 nm. There is linearity in range 5-25 µg/ml shows calibration curve with regression coefficient 0.9965. The physical constant of Felodipine was 143^0C within limits. FT-IR spectra of Felodipine show the sharp and

accurate peaks as per the standard spectra. The method validation parameters were satisfactorily results and hence this is used in estimation of Felodipine in its pure drug.

CONCLUSION

The proposed UV-visible spectrophotometric method is accurate, precise and reliable for the method validation and development study of Felodipine in bulk drug. The developed spectrophotometric method was validated for Felodipine using linearity, precision, robustness and ruggedness. The developed method is used for routine quantitative analysis in pharmaceutical preparation.

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CONFLICT OF INTEREST

Author doesn't have any conflict of interest.

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