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DEVELOPMENT AND SPECTROPHOTOMETRIC METHOD VALIDATION OF NIFEDIPINE IN SOLID DOSAGE FORM

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ABSTRACT

The simple, rapid, sensitive and specific method of spectrophotometrically was developed for the validation of nifedipine in solid pharmaceutical dosage form i.e. tablet formulation. The UV spectrum of nifedipine in methanol showed λ max at 249nm. The linearity was established in the concentration range of 10-60µg/ml for nifedipine. This method was validated for different analytical parameters such as linearity, accuracy, precision, ruggedness and robustness. The method has been shown approximate linearity over the range of 10-60µg/ml with the regression equation y=0.007x+0.022 and regression correlation coefficient r²= 0.996. However, the method was found to be highly precise with LOD (0.041) and LOQ (0.12). Considering above results the developed method can be successfully performed for the assay of nifedipine in different pharmaceutical dosage form.

KEYWORDS

Nifedipine, Spectrophotometry, Methanol, Method development and Validation.

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INTRODUCTON

Nifedipine is chemically known as dimethyl-1, 4dihydro-2, 6-dimethyl-4(2-nitrophenyl) pyridine 3, 5-dicarboxylate is a calcium channel blocker. It is used as anti-anginal agent that mainly acts as calcium channel blocker that inhibits the transmembrane influx of calcium ions into cardiac muscle cells¹⁻⁶. It is also used for treating vascular disorders such as Raynaud's phenomenon. It is mainly used in the treatment of diuretics and ACE inhibitors even though its main action is calcium channel antagonist. Previously it has been used as emergencies in hypertensive. It has very low bioavailability, thermolaible and photosensitive. After exposing the drug to light and certain

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wavelength it gets converted to a nitro phenyl pyridine derivative. Nifedipine is the common prescribed active pharmaceutical ingredient for cardiovascular disease. Even though it is highly non polar compound and get absorbed completely from GIT, mainly from jejunum, but it has very low bioavailability due to this it undergoes presystemic metabolism⁷⁻¹⁵.

The main aim of this paper is to determine spectrophotometric method of Nifedipine in pharmaceutical formulation.

MATERIAL AND METHODS

Nifedipine was taken as gift sample from J.B chemicals and pharmaceuticals pvt.ltd, India.

Methanol was used as lab grade. Nicardia Retard tablet 20mg was purchased from local retailer.

Apparatus

UV-visible spectrophotometer (systronics 2201) with 1cm quartz cells was used for all absorbance measurement, weighing balance (Shimadzu) and sonicator (Oscar ultrasonic cleaner microclean-103).

Experimental

Procedure for the determination of nifedipine

Weigh accurately 10mg of nifedipine, dissolve in 10ml of volumetric flask containing methanol and sonicate it for 10 minutes. Makeup the volume with methanol upto 10ml. Aliquots of 0.2ml, 0.4ml, 0.6ml, 0.8, 1ml were taken and diluted upto 10ml with methanol.

Procedure for assay of nifedipine in pharmaceutical formulations

An accurately 20 tablets containing nifedipine were weighed and powdered. The weighed portion of the powder equivalent to 10mg of nifedipine was then added into the 100ml of volumetric flask containing methanol and it was stirred in the sonicator for 10mins. The solution was then filtered and five aliquots were taken i.e. $20-100\mu$ g/ml and volume was made upto mark by using methanol. The solution was analyzed at 249nm wavelength.

RESULTS AND DISCUSSION¹⁶⁻²⁰ Linearity

The aliquots of different concentration such as $20-100\mu$ g/ml were taken. It shown that the linearity of nifedipine was confirmed as well as absorbance was measured. The linearity was performed on single day only. At wavelength 249nm the absorbance showed the good regression coefficient. The linearity graph was plotted against absorbance of nifedipine vs concentration of nifedipine. The regression coefficient was found to be better i.e. 0.996. Hence the parameter was found to be validated.

Accuracy

The accuracy of an analytical method is parameter that describes the closeness of the obtained test results to the theoretical value. The accuracy was performed by the standard addition method by making use of previously analyzed standard solutions. With help of these standard solutions the percentage relative standard deviation and percentage recovery were analyzed.

Range

The range of an analytical method is parameter that gives interval between lower and upper concentration limit of an analyte i.e. $20-100\mu$ g/ml.

Precision

The precision of an analytical method is parameter which is performed as inter-day and intra-day. Intraday precision was carried out in single day and inter-day precision was carried out in three days. The nifedipine was evaluated at concentration of 40μ g/ml. The percentage RSD for inter-day precision was found to be 0.518% and intra-day precision was found to be 0.2223%. The result of Inter-day and Intra-day were found be within limit i.e. NMT 2%. Hence the parameter was found to be validated.

Limit of Detection (LOD)

The limit of detection is the lowest quantity of a substance that can be detected but not necessarily quantified as an exact value. The limit of detection was found to be 0.041μ g/ml.

LOD = 3.3×Standard Deviation/ Slope

Limit of Quantification (LOQ)

The limit of quantification is defined as the lowest amount of analyte in the sample and that can be determined quantitatively by using the suitable accuracy and precision. This LOQ is used to determine the impurities and / or degradation products. The limit of quantification was found to be 0.12μ g/ml.

 $LOD = 10 \times Standard Deviation / Slope$

Ruggedness

Ruggedness of an analytical method is parameter that determines the degree of reproducibility of the test results which is performed by using same operational and environmental condition with different analyst. The percentage RSD was found to be 0.508% and 0.594%. The result of ruggedness was found within limit i.e. NMT 2%. Hence the parameter was found to be validated.

Robustness

Robustness of an analytical method is parameter that determines the small variations in parameters such as stability and temperature of an analytical solution. The percentage RSD was found to be 0.768% and 1.142%. The result of robustness was found within limit. So the parameter was found to be validated.

S.No	Concentration (µg/ml)	Absorbance		
1	20	0.176		
2	40	0.318		
3	60	0.447		
4	80	0.592		
5	100	0.768		
Table No.2: Optimization parameter for method development of Nifedipine				
S.No	Parameters	Method Values		
1	λ max	249nm		
2	Beer's law	20-100µg/ml		
3	Regression equation $(Y = mx + c)$	y = 0.007x + 0.022		
4	Correlation coefficient (r)	0.996		
5	Intercept	0.022		
6	Slope	0.007		
7	LOD (µg/ml)	0.041		
8	LOQ (µg/ml)	0.12		
Table No.3: Intra-day Precision				
S.No	Concentration(µg/ml)	Absorbance		
1	40	0.318		
2	40	0.317		
3	40	0.318		
4	40	0.319		
5	40	0.318		
6	40	0.315		
SD		0.00070		
%RSD	-	0.2223%		

 Table No.1: Results of linearity

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Table No.4: Inter-day precision			
S.No	Concentration(µg/ml)	Absorbance Day1	Absorbance Day2
1	40	0.318	0.317
2	40	0.317	0.314
3	40	0.318	0.314
4	40	0.319	0.315
5	40	0.318	0.316
6	40	0.315	0.318
SD		0.00070	0.0013
%RSD		0.22236%	0.518%

Table	No 4.	Inter-day	nrecision
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Table No.5: Robustness

Wavelength	249nm	254nm
Concentration(µg/ml)	$15(\mu g/ml)$	$15(\mu g/ml)$
Absorbance	0.152	0.151
-	0.151	0.153
-	0.153	0.152
-	0.154	0.154
-	0.152	0.156
-	0.151	0.154
SD	0.001169	0.001751
%RSD	0.768266%	1.14208%

Table No.6: Ruggedness

S.No	Concentration (µg/ml)	Absorbance Analyst 1	Absorbance Analyst 2
1	20	0.176	0.177
2	20	0.177	0.178
3	20	0.175	0.175
4	20	0.177	0.176
5	20	0.175	0.177
6	20	0.176	0.176
7	SD	0.0009	0.0014
/	%RSD	0.508%	0.594%



Figure No.1: Structure of Nifedipine

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Figure No.2: Calibration curve of Nifedipine

CONCLUSION

Based on the above results, obtained from analysis, it can be concluded that method has linear response over the range of 20-100 μ g/ml for Nifedipine. The results of analysis of formulation by the proposed method were found to be accurate, precise, economic and less time consuming with sensitivity. This method can be easily applied for the analysis of Nifedipine in dosage forms.

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CONFLICT OF INTEREST

We declare that we have no conflict of interest.

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