Research Article **ISSN: 2349 – 7106**

Asian Journal of Research in Chemistry and Pharmaceutical Sciences

Journal home page: www.ajrcps.com

https://doi.org/10.36673/AJRCPS.2020.v08.i01.A04

METHOD DEVELOPMENT AND VALIDATION OF AMOROLFINE IN BULK AND ITS SEMISOLID DOSAGE FORM BY VISIBLE SPECTROPHOTOMETRY

Vinod Matole*[1], Smita Kumbhar[1], Yogesh Thorat[1], Saili Madur[1], Smeeta Patil[1], Avinash Birajdar[1], Anita Shegaonkar[1]

[1*]Department of Pharmaceutical Quality Assurance, D.S.T.S. Mandals College of Pharmacy, Solapur-413004, Maharashtra, India.

ABSTRACT

Objective: The present study was undertaken to develop a rapid simple specific accurate, precise, robust and economic visible spectrophotometric method for determining the Amorolfine in semisolid dosage form. **Method:** The Visible spectrophotometric method was performed at maximum wavelength 542nm by using Methanol and DMF as a solvent. The nethod was validated by following the analytical performance parameter suggested by ICH which include accuracy, precision, linearity, robustness, LOD, LOQ and Specificity. **Result:** The drug obeys the beer's lamberts law in the concentration range of 10-60µg/ml. It exhibits the good coefficient correlation (0.9992) and excellent mean recovery. The % recovery for precision was found within limit i.e. less than 2% and accuracy gave results within limit i.e. 97-103%. The developed method was suitable and specific to analysis of Amorolfine even in the presence of excipients. **Conclusion:** The obtained results proved that the validated method can be employed for routine analysis of Amorolfine in bulk as well as in the Cream.

KEYWORDS

Amorolfine, Chloranillic acid, Methanol, DMF, Visible Spectrophotometry, Method development and Validation.

Author for Correspondence:

Vinod Matole,
Department of Pharmaceutical Quality Assurance,
D.S.T.S. Mandals College of Pharmacy,
Solapur-413004, Maharashtra, India.

Email: matole7414@gmail.com

INTRODUCTON

Visible Spectroscopy is most widely used method in the analytical chemistry. It is also called as colorimetry. Amorolfine is used as a morphine antifungal agent that inhibit the fungal enzymes D14 reductase and D7-D8 isomerase. This inhibition affects fungal sterol synthesis pathway, by depletingergosterol and causing ignosterol to accumulate in the fungal cytoplasmic membrane[1-2]. Amorolfine is Soluble in methanol, ethanol, DMF and DMSO. IUPAC name of Amorolfine is (2R,6S)

2, 6- dimethyl-4-(2-{[4-(2-methylbutan-2-yl) phenyl] methyl} propyl) morphine and its chemical formula is $C_{21}H_{35}NO^{3}$.

MATERIAL AND METHODS

Materials

Amorolfine was taken as a gift sample from Optimus Pharma Pvt. Ltd., Bangalore, Chloranillic acid and DMF were taken from Lab grade basis.

Instruments

UV Visible Double beam Spectrophotometer (Systronics 2201), with 1cm quartz cuvettes were used for analysis of absorbance.

EXPERIMENTAL

Preparation of Chloranillic acid

Chloranillic acid (0.3% w/v) was dissolved in methanol and Make up the volume upto 10ml using DMF.

Preparation of Standard Stock Solution

For standard stock solution, weigh accurately 10mg of drug and dissolved in methanol and vortexed it for 2min and make up the volume upto 10ml using DMF.

Procedure for Plotting Calibration Curve

From standard stock, pipette out (0.1, 0.2, 0.3, 04, 05 and 0.6) ml separately and add 0.1ml in each flask 0.3% w/v of Chloranillic acid dissolved it in methanol and make up the volume upto 10ml using DMF.

Assay of Amorolfine in Pharmaceutical dosage form

For Assay weigh accurately 10mg equivalent amount of cream and was dissolved in methanol and make up the volume upto 10ml using DMF and vortex it for 2 minutes and sonicate it for 10 minutes. From above solution pipette out 0.1-0.6ml and add in each flask separately and dissolved in methanol, add 0.1ml 0.3% w/v of Chloranillic acid in each flask and make up the volume upto 10ml using DMF.

RESULTS AND DISCUSSION

The absorption Spectrum shows the result of wavelength at 542nm.

The Proposed method Visible Spectrophotometry of Amorolfine was validated as per ICH guidelines[4-5].

Linearity

Linearity is defined as "The obtained results are closeness to the true values in the analytes is called linearity." The linearity was found at concentration 10-60µg/ml. and the regression coefficient was found to be 0.9992.

Assay

Assay was performed using Amlostar Cream. The assay was matched at concentration 50µg/ml. The % purity was found to be 99.3%.

Range

The parameter range was found in 10-60µg/ml. The range is the difference between upper and lower limit of analyte.

Accuracy

The parameter accuracy is the extent to which the experimental results deviates from the expected results and it is a measure of the trueness of the analytical method. Accuracy may be reported as.

Precision

Precision is divided into two types intraday and Interday precision. Intraday precision was performed in one day and Interday precision i.e. at concentration 30µg/ml were performed in different days. The % RSD of Intraday and Interday was found within limit that is less than 2%. Hence the Parameter was found to be validated.

LOD and LOQ

By using Standard deviation and Slope, LOD and LOQ were determined. Which are as follows.

Robustness

The change in Wavelength i.e. 542nm and 554nm as well as change in Concentration i.e. 12µg/ml, there was no effect on the Absorbance. The obtained results are as follows.

Table No.1: Optimization Parameters of Amorolfine

S.No	Parameters	Method values
1	Maximum Wavelength	542nm
2	Beer's law	10-60µg/ml
3	Correlation Coefficient	0.9992
4	Regression Equation	0.0181x - 0.0885
5	Slope (m)	0.0181
6	Intercept	0.0885

Table No.2: Results of linearity

S.No	Concentration (µg/ml)	Absorbance
1	10	0.104
2	20	0.262
3	30	0.449
4	40	0.631
5	50	0.829
6	60	0.994

Table No.3: Assay of Amorolfine

S.No	Formulation	Labeled Amount	Amount obtained	% recovery
1	Amlostar cream	0.25%	0.243%	99.3%

Table No.4: Accuracy of Amorolfine

S.No	Name of Drug	Recovery Level in %	Concentration	Amount Recovered	% recovery with SD
1	Ketoconazole	80	30µg/ml	30.03	100.03±0.7
		100	40µg/ml	40.04	99.04±0.6
		120	50µg/ml	50.05	100.5±0.5

Table No.5: Results of Intraday Precision

S.No	Concentration	Absorbance
1	30µg/ml	0.449
2		0.447
3		0.445
4		0.448
5		0.449
6		0.448
7	SD	0.001506
8	%RSD	0.336309%

Table No.6: Results of Interday Precision

S.No	Concentration	Absorbance Day1	Absorbance Day2
1	30µg/ml	0.449	0.447
2		0.447	0.445
3		0.445	0.447
4		0.448	0.448
5		0.449	0.449
6		0.448	0.446
7	SD	0.001506	0.001414
8	%RSD	0.336309%	0.316379%

Table No.7: Results of LOD and LOQ

LOD	1.69µg/ml
LOQ	5.65µg/ml

Table No.8: Results of Robustness

Wavelength	542nm	554nm
Concentration	12µg/ml	12µg/ml
Absorbance	0.131	0.133
	0.132	0.131
	0.131	0.133
Average	0.13313	0.13233

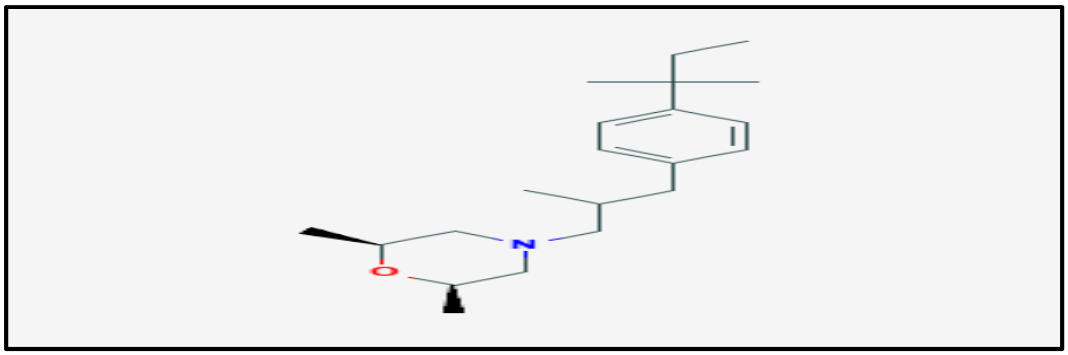

Figure No.1: Structure of Amorolfine

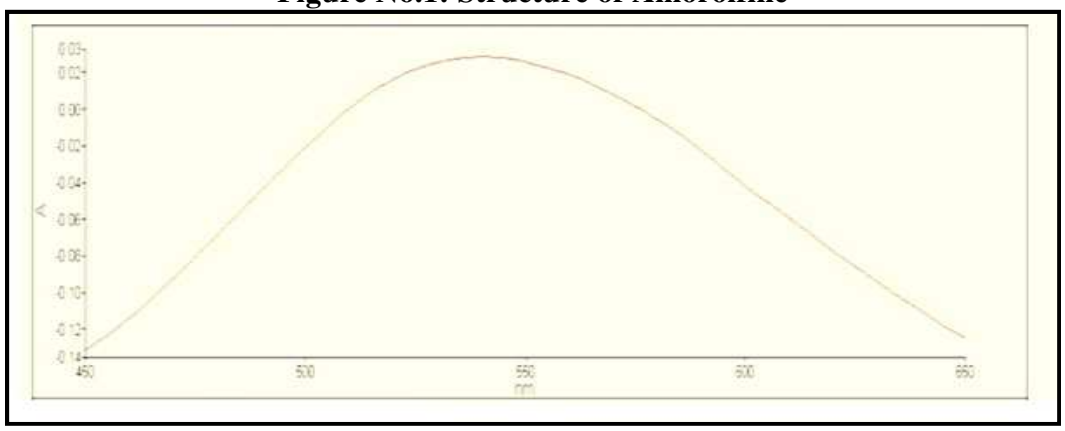

Figure No.2: Visible Spectrum of Amorolfine

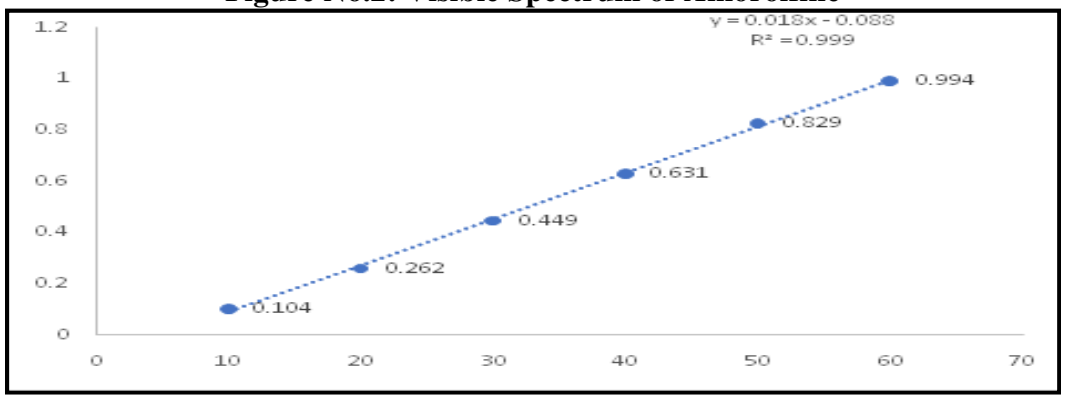

Figure No.3: Calibration curve of Amorolfine

CONCLUSION

The Visible Spectrophotometric method was developed and validated thoroughly for the quantitative determination of Amorolfine in pure and its semisolid dosage form. The proposed method was simple, linear, accurate, specific, precise and robust and gives an acceptable recovery of the analyte, which can be easily applied to the semisolid dosage form of Amorolfine i.e. Amlostar cream.

ACKNOWLEDGEMENT

The authors wish to express their sincere gratitude to Department of Pharmaceutical Quality Assurance, D.S.T.S. Mandals College of Pharmacy, Solapur-413004, Maharashtra, India for providing necessary facilities to carry out this research work.

CONFLICT OF INTEREST

We declare that we have no conflict of interest.

BIBLIOGRAPHY

1. Aparna V, Rao G. Spectrophotometric development for the determination of amorolfine, *Oriental J. of chemistry,* 26(3), 2010, 1207-1209.
2. Aparna A, Rao G. Spectrophotometric methods for the determination of amorolfine, *Int. J. Chem. Sci.,* 8(4), 2010, 2737-2376.
3. Soto C *et al.* A new Kinetic spectrophotometric Method for the Quantitation of Amorolfine, *J of analytical methods in chemistry,* 12(1), 2017, 1-8.
4. Verma V, Kumar S. Ketoconazole HPLC Method Development and validation: A novel Approach, *Int. Research journal of Pharmacy,* 8(8), 2012, 74-81.
5. $ICHQ_2$ (R_1) guidelines, Validation of analytical procedures text and methodology, *International Conference on Harmonization,* 1996, 1-16.

Please cite this article in press as: Vinod Matole *et al.* Method development and validation of amorolfine in bulk and its semisolid dosage form by visible spectrophotometry, *Asian Journal of Research in Chemistry and Pharmaceutical Sciences,* 8(1), 2020, 17-21.