WORLD JOURNAL OF PHARMACEUTICAL RESEARCH
SJIF Impact Factor 8.084
Volume 12, Issue 16, 834-841. Research Article ISSN 2277– 7105

DEVELOPMENT AND VALIDATION OF UV-SPECTROSCOPIC METHOD FOR THE ESTIMATION OF OXETACAINE IN BULK AND PHARMACEUTICAL DOSAGE FORM

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Article Received on
01 August 2023,

Revised on 21 August 2023,
Accepted on 11 Sept. 2023

DOI: 10.20959/wjpr202316-29669

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ABSTRACT

A new, trouble-free and sensitive Spectrophotometric method in ultraviolet region has been developed for the estimation of oxetacaine in bulk and pharmaceutical dosage form. It was the main objective of this study for know about the oxetacaine should be still any research there is no developmental method in UV – spectroscopy. It is the first article to reveal the method development in spectroscopy for oxetacaine. Quantitation was based on peak area at 206 nm. The method obeys the Beer's law in the concentration range from 1.5 – 5.5 µg/ml. The linearity was observed in the above concentration. Here we using only methanol as a solvent to reduce our cost for double solvent using in estimation. The limit of detection for oxetacaine was obtained in satisfactory limits such as 0.051µg/ml. The limit of quantification of oxetacaine was obtained in reasonable limits such as 0.156µg/ml. The percent recovery of oxetacaine was found to be 99. 56%. The percentage relative standard deviation for the intraday and interday precision was obtained in the range of 1.191 & 1.349%. The obtained results were confirmed that the proposed method is specific, rapid, accurate, reproducible and suitable for the routine determination of

oxetacaine. And also the above proposed method was validated according to the ICH guidelines.

KEYWORDS: Oxetacaine, UV – spectroscopic calibration curve method, Methanol, validation.

1. INTRODUCTION

Oxetacaine (OXT) or 2,2 – (2-hydroxyl ethyl amino) bis[N- 1,1- dimethyl- 2- phenylethyl – N- methylacetamide], a glycine amide resembling lidocaine, is a potent safe local anaesthetic agent. And another topical application, it provides prolonged anaesthetic action on mucus membrane. It includes ability to remain unmanaged event at the pH of 1, unlike most of the other local anaesthetics. It contact with a receptor presented within the voltage gated sodium channel and raises the entrance of channel opening in that way sodium ions permeability fails to boost in response to an impulse or stimulus and also exceeding by far the potency of cocaine, procaine or dibucaine.[1] It was given by orally in combination with an antacid for prolonged relief of pain associated with peptic ulcer diseases like esophagitis and gives an anti-spasmodic action on smooth muscle. It is also called as oxethazine. The British pharmacopoeia analysed oxetacaine by a potentiometric method and titration with 0.1 M perchloric acid. The world Anti– Doping agency has forbidden the following two metabolites: mephenterdrine and phenterminum. These two are the metabolites of oxetacaine. It is moreover used for Remittal hernia. Alsoone more Gas Chromatography – Mass spectroscopy method to structurally establish the contents include the parent drug and also their metabolites in urine and the concomitant administration of other drugs to determine the effect of inhibition and induction of drug metabolism. And it is also a source for athletes. It has the half life to be 1 hour. It was only in the form of suspension.[2,3,4] Finally, we complete it. It can be analysed and the method was developed under UV – spectroscopic method and perform better validation as per International Council on Harmonisation (ICH) guidelines.

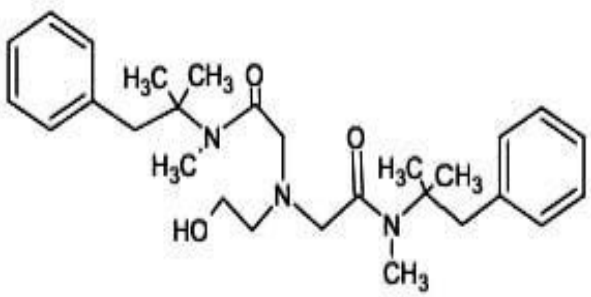

Fig. 1: Structure of oxetacaine.

2. MATERIALS AND EXPERIMENTAL WORK

2.1. Instrumentation

Shimadzu 1900i – The double beam UV – Visible spectrophotometer, then its absorption spectra of a test and reference solutions were recorded in 1 cm quartz cells from the range of 200 – 400 nm.

2.2. Source of drug

Oxetacaine was obtained in both as a raw material and dosage form. In this the raw material was provided as a kind of gift sample from SAI MIRRA INNOPHARM PVT. LTD., Chennai and the dosage form (Suspension - 200 ml) was withdrawn from the market. Better solubility for above drug using only Methanol (Analytical Grade) as a solvent in this method. It was obtained from THE PRECISION SCIENTIFIC CO., Coimbatore.

2.3. Methodology

2.3.1 Choice of solvents

The methanol was selected as the solvent for Oxetacaine. The spectrum was clearly observed by using this solvent. It was insoluble in water and so we have to select the solvent (methanol) to better solubility by even taking 10 mg of oxetacaine.

2.3.1. Preparation of stock solution

Accurately weighed quantity of oxetacaine (OXT – 0.01 g) was dissolved in 10 ml Methanol to get a primary stock solution (1000 µg / ml). From this above solution we make a secondary stock solution (100 µg / ml) by dissolving of taking 1 ml from above solution with making 10 ml methanol. Then finally we make serial dilutions of 1.5 – 5.5 µg /ml solution from the above secondary make up with 10 ml methanol. And get this concentration was scanned between 200 – 400 nm. It gives better absorbance and get a spectrum of oxetacaine. It was found to be 206 nm.

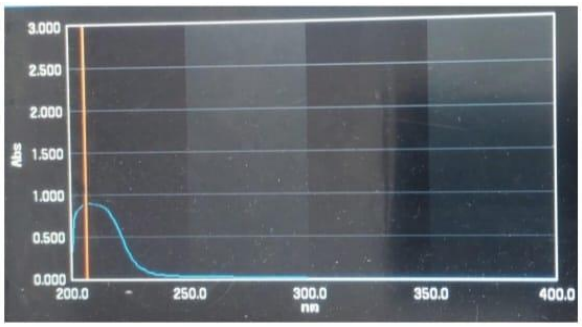

Fig. 2: Gamut of oxetacaine.

2.3.2. Preparation of calibration graph

From the standard stock solution 1.5 – 5.5 ml were taken and transferred into chain of 10 ml volumetric flask and make up with the solvent of methanol. Then determine the absorbance from the above concentration to be measured and the calibration curve was obtained by plotting absorbance Vs concentration. It was linear with the concentration range of 1.5 – 5.5 µg / ml and obtained with regression coefficient value.

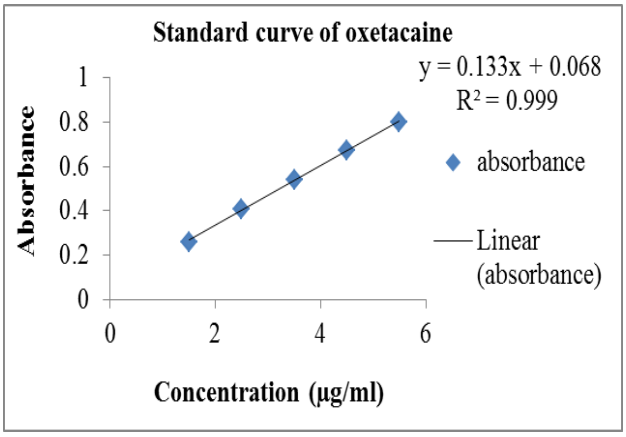

Fig. 3: Standard curve of oxetacaine.

2.3.3. Preparation of sample solution

The weight equivalent to 5 ml of oxetacaine suspension was weighed and transferred to a 100 ml volumetric flask, dissolved the content with methanol in 100 ml by using Sonicator for 10 minutes and filter it thoroughly by using Whatman filter paper grade no. 1. Then take a clear solution and make up the solution with diluent (methanol) to obtain a concentration of 100µg / ml of oxetacaine. The above solution was further diluted with the diluent to produce the concentration of 10, 30 and 50 µg / ml. Finally absorbance was measured at UV region with regression equation.

3. Validation

The validation parameters were performed for above developed method in compliance with the ICH Q2B recommended conditions. The parameters are carried out by linearity, accuracy, precision, recovery studies and LOD & LOQ.

3.1 Linearity

To ensure linearity, perform calibration curve for different concentrations from 1.5 – 5.5 µg / ml. There should be prepared from stock solution of oxetacaine. The curve was obtained by

plotting the concentration Vs absorbance. The slope and intercept values were recorded with respect to their wavelength.

3.2 Accuracy

The accuracy of the method was determined and suggested technique's recovery study was resolved by the standard addition method at 80 %, 90 % and 120 %. The stock solution of standard and the sample was prepared. Pipette out 0.5 ml of the standard into three standard volumetric flasks and add the sample 0.3, 0.5, 0.7 ml into above pipette out volumetric flasks and makeup the volume up to 10 ml with methanol. The solution was measured under a UV spectrometer and the % recovery was determined.

3.3 LOD

Limit of detection was resolute from the calibration curve values of standard deviation and slope intercept. LOD is calculated from the rule,

LOD = 3.3 S.D/Slope

It should be the least concentration of the analyte that gives a quantifiable response.

3.4 LOQ

Limit of quantification was resolute from the calibration curve value of standard deviation and slope intercept. LOQ is calculated from the rule,

LOQ = 10 S.D / Slope

It should be the least concentration of the analyte that gives a quantifiable response.

3.5 Robustness and Ruggedness

The ruggedness and robustness of the methods were studied by changing the experimental conditions (operators, source of reagents) and optimised conditions (pH, temperature, etc..,).

3.6 Assay

The drug content present in the formulation was analysed and determined by statistical method.

4 RESULT AND DISCUSSION

Here, the above method was validated accurately and the getting validations results are discussed below, they are correlation coefficient (r^2) value obtained from calibration graph was getting to be 0. 999 which indicate that the absorbance was linear with the concentration range of 1 – 5 µg / ml and also the regression equation was found to be y = 0.143x + 0.009 (y

= mx + c) are shown in table.1&2. The percentage RSD value was getting to be 1. 4538 are shown in table.2. The limit of detection (LOD) and limit of quantification (LOQ) was getting to be 0.051 µg / ml and 0.156 µg / ml was revealed in table.2. The percentage RSD value for intraday precision and inter day precision was getting to be 1.349 and 1.191 were revealed in table.3&4. The accuracy should be determined by recovery studies, it has the % RSD value was getting to be 0.741 was presented in table.5. The percentage RSD value for assay was getting to be 0.302 by performing SUCRACYZ – O was revealed in table.6. Therefore, the method was created to be accurate should be proved.

Table 1: Linearity of oxetacaine.

Concentration (µg/ml)	Absorbance
1.5	0.261
2.5	0.409
3.5	0.542
4.5	0.674
5.5	0.798

Table 2: Linearity data shows statistical data at the selected wavelength.

Wavelength (nm)	Regression equation	r^2	LOD (µg/ml)	LOQ (µg/ml)	% RSD
206	y = 0.1339x + 0.0682	0.999	0.051	0.156	1.4538

Table 3: Data for intraday precision.

Concentration (µg/ml)	Absorbance
1.5	0.367
2.5	0.374
3.5	0.378
4.5	0.371
5.5	0.381
Average	**0.373**
%RSD	**1.349**

Table 4: Data for inter day precision.

Concentration (µg/ml)	Absorbance
1.5	0.367
2.5	0.378
3.5	0.371
4.5	0.379
5.5	0.374
Average	**0.374**
% RSD	**1.191**

Table 5: Recovery studies of oxetacaine.

Wavelength (nm)	Amount present (µg/ml)	Amount added (µg/ml)	Absorbance	Amount recovered (µg/ml)	% Recovery
206	10	8	0.385	7.87	98.38
		10	0.625	10.11	101.10
		12	0.520	11.96	99.67
	10	8	0.397	7.97	99.63
		10	0.452	10.05	100.50
		12	0.673	11.97	99.75
	10	8	0.443	7.96	99.50
		10	0.567	9.97	99.70
		12	0.768	11.95	99.58
Average					99.75
% RSD					0.741

Table 6: Assay for oxetacaine.

Brand name	Label claim (ml)	Amount estimated	% Assay
SUCRACYZ – O	150	149.8	99.87
	150	149.8	99.53
	150	149.9	99.27
	Average		**99.56**
	%RSD		**0.302**

5 CONCLUSION

We conclude that the UV spectroscopic method have been developed for the estimation of oxetacaine in bulk and pharmaceutical dosage form. In this technique, methanol was used as solvent. The standard solution of oxetacaine was scanned at 206 nm and the Beer's law limit was 1.5 – 5.5 µg/ml. The correlation coefficient value, r^2 = 0.999, LOD= 0.051, LOQ= 0.156 and the percentage purity of oxetacaine formulation were found to be 99.56%. The proposed method quantitatively evaluated in terms of linearity, accuracy, precision, LOD, LOQ and recovery. All these factors lead to the conclusion that the proposed UV – spectroscopic method was simple, accurate, precise, sensitive and cost effective. This method was adopted for the use of economical and easily available and for the UV detection. Finally this method was so economical for routine use. The above developed technique was validated by means of ICH guiding principles. The observed values are getting within the respected limits.

6 ACKNOWLEDGEMENT

It gives us an enormous pleasure to acknowledge, we grab the occasion to express our unfeigned thanks to our respected principal Dr. K. B. Ilango M. pharm., Ph.D., Shree Venkateshwara College of Paramedical Sciences, Gobi – Tamil Nadu, for his invariable

support and encouragement. Without his supervision it would have been utterly unfeasible to bring out the work in this manner and also heartfelt thanks to our venerated and most prized staffs and non teaching staffs for their valuable support during this research work.

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