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Research Article

UV Spectrophotometric Estimation of Satranidazole in Periodontal Gels

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ABSTRACT

A simple, precise and economical UV method has been developed for the estimation of Satranidazole in periodontal gels. Satranidazole has the absorbance maxima at 319 nm in McIlvaine buffer pH 6.6.Beer's law was obeyed in the range 1–40µg/ml with correlation coefficient (n = 6) ≥ 0.9996.The proposed methods were successfully applied for the simultaneousdetermination of Satranidazolein gel dosage form. The developed method was validated for accuracy and precision. Percent recovery for Satranidazole was found in the range of 98.14 % to 100.68 %. A high percentage recovery indicates the high accuracy of the method. This method has the advantage of quicker turnaround time of sample analysis and less cost involved. It can act as an alternative for HPLC method for the routine use in quality control laboratories of pharmaceutical industry for estimation of Satranidazole.

Keywords: Gel, *In-vitro*dissolution, Satranidazole, UV spectrophotometry.

INTRODUCTION

Satranidazole,1-methylsulfonyl-3-(1-methyl-5-nitro-2-imidazolyl)-2-imidazolidinone[1],is used as anantiprotozoaland antibacterial agent in the treatment of amoebiasis and periodontitis [2].Literature survey revealed that Satranidazole(SZ) isestimated byHPTLC[3, 4], HPLC [5], colorimetric methods[6,7,and 8],HPLC method for combination of SZ and Ofloxacin[9], UV method for SZ tablet [10], UV method for combination of SZ and Ofloxacin[11] and by RP HPLC [12]technique. There is no reported literature for UV estimation of Satranidazole in gel formulation; though one method has been reported onlyfor estimation of SZ intablet formulation[10]. Periodontal gel formulation of SZ has been developed in our laboratory [2] in Department of Pharmaceutics, Institute of Technology, Banaras Hindu University, Varanasi, India and analyzed by UV method. Direct UV/VIS spectrophotometric determination of drug contentis the traditional analytical method for dissolution testing [13] and has been known for its simplicity, selectivity andsensitivity. A compound will exhibit absorption in the UV region if it contains chromophore groups like aromatic nitro, azoxy, nitroso,carbonyl,or azo groups[13]. Aim of present study is to validate and elaborate UVmethod for estimation of Satranidazolein gel formulations and demonstrate good accuracy, simplicity, precision and economy.

MATERIALS AND METHODS

Satranidazole was obtained as a gift sample from Alkem Laboratories Pvt. Ltd., Mumbai, India. Sodiumcarboxymethylcellulose(SCMC) was procured from S.D. fine-chem. Ltd., India and Carbopol934P(CB934P) was sourced from LobaChemie Pvt. Ltd., Mumbai, India. Disodium hydrogen orthophosphate anhydrous obtained from Qualigens fine chemicals, Mumbaiand citric acid monohydrate sourced from Merck Ltd., Mumbai. Purified water was used as asolvent. A double-beam UV-Vis spectrophotometer (Shimadzu, Japan) model UV - 1800, with a fixed slit width (2 nm) using 1.0 cm quartz cells was used for all absorbance measurements.

Formulation of Satranidazole Gel: Satranidazole periodontal gel was prepared in our laboratory in Department of Pharmaceutics, Institute of Technology, Banaras Hindu University, using a pre-optimized formula (Table 1) using Sodiumcarboxymethylcellulose (SCMC) and Carbopol934P (CB934P). Satranidazole was found to be clinically effective at concentration 0.25% as per our previous work [2]. The gel formulation was prepared by dissolving SZ in 25 ml of McIlvaine buffer pH 6.6 and adding it to a solution of carbopol 934P in 60 ml of McIlvaine buffer pH 6.6[2]. Added SCMC slowly under

Table 1: Batch Formula for Manufacturing of Periodontal Gel

Ingredients	Quantity per batch (in gm)
Satranidazole	0.25
Sodiumcarboxymethylcellulose	2.50
Carbopol 934P	1.00
McIlvaine buffer pH 6.6	96.25
Total Weight	100.00

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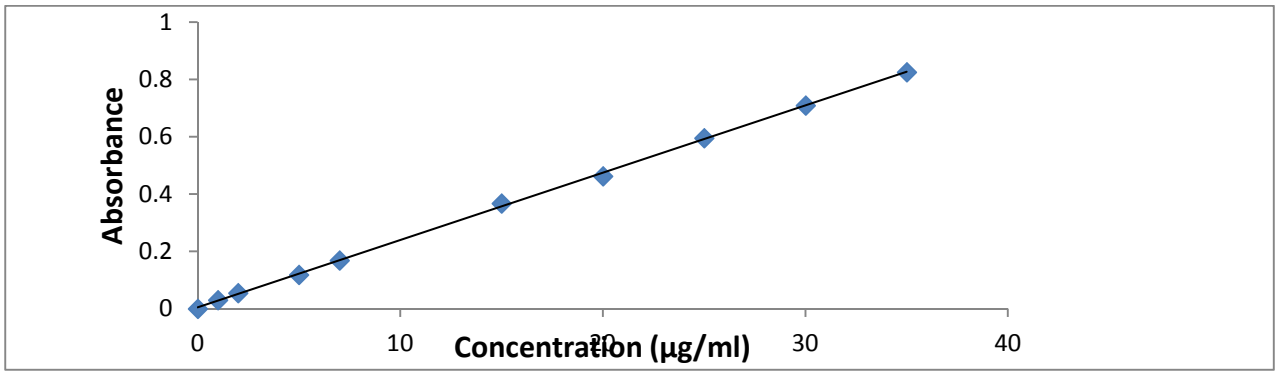

Figure 1: Standard Calibration Curve of Satranidazole in McIlvaine buffer pH 6.6

Table 2: Dissolution Profile of SZ Containing Periodontal Gel

Time (minutes)	Cumulative Percent Release (CPR)
30	22
60	41
120	57
180	68
240	74
300	79
360	82
420	84

continuous magnetic stirring at 100 rpm and the final volume was made up to 100 ml with McIlvaine buffer pH 6.6. The prepared gel was kept for 24 hours at room temperature for complete polymer dissolution.

Preparation of McIlvaine Buffer pH 6.6: 26.05 g of disodium hydrogen orthophosphate anhydrous and 2.85 g of citric acid monohydrate were mixed with about 50 ml of distilled water. The volume was then made up to 500 ml with distilled water.

Construction of Standard Calibration Curve: Accurately weighed 10 mg of satranidazole was dissolved in McIlvaine buffer pH 6.6 by heating it for a few minutes. The volume was made up to 100 ml to give a stock solution of 100µg/ml concentration. From this stock solution, graded dilutions in the concentration range of 1 to 35µg/ml were prepared and scanned in the spectrum modefrom 400 nm to 200 nm. The λmaxof SZ was found to be 319 nm in McIlvaine buffer pH 6.6. The absorbance of prepared standard solution was measured and plotted against concentration to get a standard calibration curve (figure 1).By using the calibrationcurve, the concentration of the sample solution wasdetermined.

Analytical Method Validation: The recovery studies were carried out at different level of concentration by spiking a known concentration of SZ to the placebo sample and contents were analyzed by UV spectrophotometry. The methods were validated as per ICH Q 2 (B) guidelines [14] for parameter like accuracy, precision, linearity andrange.

Accuracy was ascertained on the basic of recovery studies. Precision was studied by analyzing nine replicates [14] of sample solution and concentrations were calculated. Ruggedness was established by carrying out experiment at different time within a day (intraday), different day (interday) and by differentanalyst. Linearity and range were determined byanalyzing 80-120% of test concentrations of each drug [11].

Dissolution Study: Dissolution study was conducted using a dialysis bag and McIlvaine buffer pH 6.6 as the dissolution medium [2]. Periodontal gel (equivalent to 2.5 mg of SZ) was taken in the center of a hollow cylindrical dialysis membrane, folded it and hermetically sealed from both the ends. This dialysis bag was hanged with a wire in a 100 ml beaker. The entire system was kept at 37.0 ± 0.5°C with continuous magnetic stirring at 100 rpm (Figure 2). Quantity of dissolution media was taken 40 ml. Sampling aliquots of 5 ml were withdrawn at 30, 60, 120, 180, 240, 300, 360 and 420 minutesand replaced with an equal volume of the fresh medium to maintain a constant total volume. After the end of each test time, samples aliquots were filtered and diluted in dissolution medium and quantified using UV spectrophotometer at 319 nm. Sink conditions were maintained for release studies (C_1 < Cs X 0.2) [2].

C_1 = Final concentration of satranidazole in medium after the complete release of the drug in the McIlvaine buffer pH 6.6

Cs = Saturation solubility of satranidazole in the McIlvaine buffer pH 6.6

RESULTS AND DISCUSSION

In this study, periodontal gels of Satranidazole were prepared; the saturation solubility of drug was found to be 416.54±35.64µg/ml[2]; the dissolution study was conducted in 40 ml of McIlvaine buffer pH 6.6 and drug content was analyzed by UV method at 319 nm (Table 2 and Figure 3). As per calculation $C_1$was 62.5µg/ml and Cs X 0.2 was 83.308µg/ml[2]. Dissolution media 40 ml was able to establish the sink conditions.

The absorption spectrum of Satranidazole was measured in the range 200–400 nm. The methods discussed in the present work provide aconvenient and accurate way for analysis of Satranidazole in pharmaceutical gels. The method validation parameters were as: R^2 = 0.9996, Accuracy = 99.88 ± 2.03, Precision =2.06, Range = 1 to 35 µg/ml, Linearity = 1 to 40µg/ml .Data from the regression line was used to provide mathematical estimates of the degree of linearity [14].The regression equation was calculated and found to be y = 0.0235x + 0.0043.The

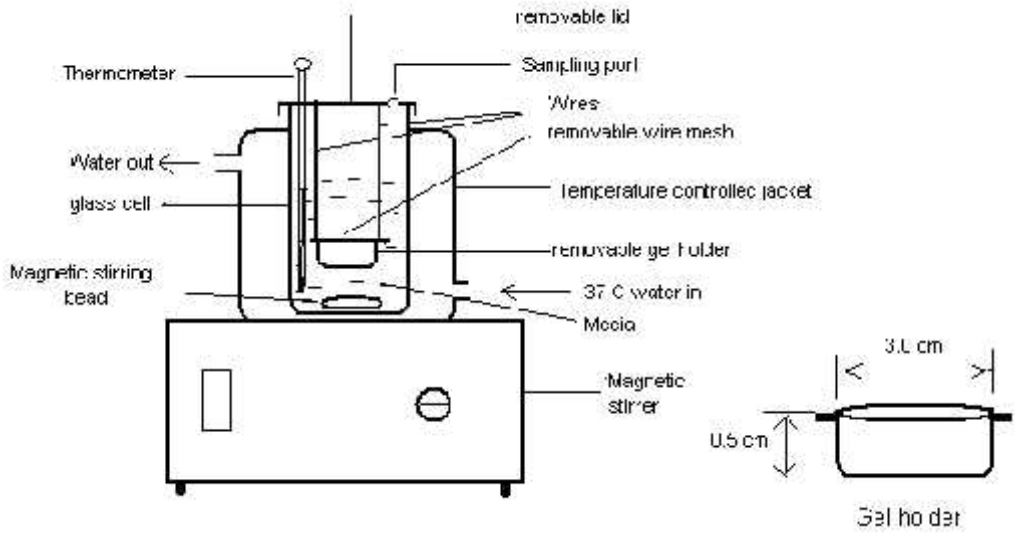

Figure 2: Schematic Diagram of DissolutionStudy Apparatus

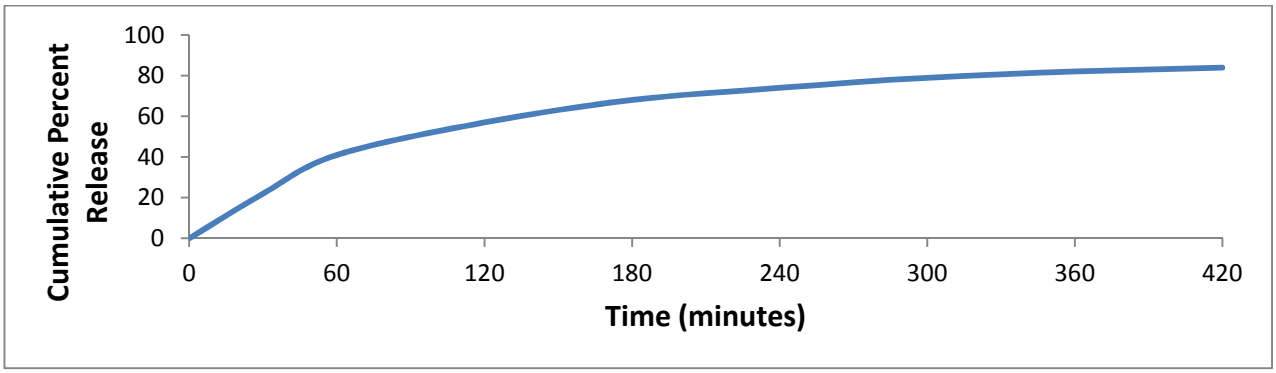

Figure 3: Release Profiles of SZ Containing Periodontal Gel

detection limit (LOD) for the proposed methods was calculated using the following equation:

LOD = 3SD / k

Where SD is the standard deviation of replicate determination values under the same conditions as for the sample analysis in the absence of the analyst and k is the sensitivity, namely the slope of the calibration graph. In accordance with the formula, the detection limits were found to be 0.846 µg/ml. The limit of quantization, LOQ, is defined as:

LOQ = 10 SD / k

According to this equation, the limit of quantization was found to 2.564µg/ml. The linearity of calibration graphs was proved by the high values of the correlation coefficient (R^2) and the small values of the y-intercepts of the regression equations. Assay of Satranidazole gel was found to be 99.98%. Percent label claim for SZ in content uniformity analysis was found in the range of 98.93 % to 100.87 %. Accuracy of proposed methods was ascertained by recovery studies and the results are expressed as %recovery. Percent recovery for SZ was found in the range of 98.14 % to 100.68 %. A high percentage recovery indicates the high accuracy of the method. Values of standard deviation and coefficient of variation was satisfactorily low indicating the accuracy of the methods. Result of percentage recovery and placebo interference shows that the method was not affected by the presence of common excipients [13].Thus the developed method can be easily used for the routine analysis of Satranidazole in pharmaceutical gels.

Spectrophotometric analysis is of major interest in the field of analytical chemistry since it offers distinct possibility in the assay of a particular component in pharmaceutical formulations. The proposed method does not involve complex procedural steps and do not take more operator time and expertise like HPLC and other methods. The proposed UV method could be considered superior in comparison with the previously reported methods, in terms of simplicity, rapidity, sensitivity, expense and no placebo interference; especially with those based on chromatography [3,4,5, 9,12] or other reported spectrophotometric methods [10, 11]. The chemicals, reagents and instruments used in the proposed methods are cheaper, readily available and the procedures do not involve any critical reaction conditions or tedious sample preparation. The method is unaffected by slight variations in experimental conditions such as time, reagent concentration or temperature. The UV method discussed here gave results with good accuracy to permit determination of low concentrations of Satranidazole. The proposed method can be employed for routine quality control analysis of Satranidazole in periodontal gel formulations in Quality Control laboratories of

pharmaceutical industry. Though full-fledged analytical method validation as per ICH guideline [14] need to be performed to demonstrate robustness and system suitability of the proposed method.

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