DEVELOPMENT AND VALIDATION OF UV SPECTROPHOTOMETRIC METHOD FOR QUANTITATIVE ESTIMATION OF BALSALAZIDE FROM CAPSULE FORMULATION

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ABSTRACT

Balsalazide is an anti-inflammatory drug used in the treatment of inflammatory bowel disease. It is usually administered as the disodium salt. New, simple and sensitive spectrophotometric methods for the determination of balsalazide have been developed for the quantitative estimation of from balsalazide capsule dosage form. The method was developed and based on the solubility of balsalazide in 0.1 N NaOH. The drug showed maximum absorbance at 454 nm and linearity (Lambert Beer’s Range) was found in concentration range of 10-100 μg/ml and the standard curve equation was found to be y = 0.015x – 0.015 and R² value 0.998. The results of analysis were validated statistically and by recovery studies. The result of analysis was validated as per ICH guidelines and this method can be used for the routine analysis of balsalazide formulation.

KEYWORDS: Balsalazide, 0.1 N NaOH, UV method, Validation.

INTRODUCTION

Balsalazide disodium[1-2] is chemically (E)-5-[[4-[[2-carboxyethyl]amino][carboxyl][phenyl]azo]-2-hydroxy benzoic acid, disodium dihydrate. It is a prodrug that is enzymatically cleaved in the colon to produce mesalamine (5-aminosalicylic acid), an anti-inflammatory drug. It is used in the treatment of mild to moderate ulcerative colitis.[3-5] Balsalazide disodium capsules contain granules of Balsalazide disodium, which are insoluble in acid and designed to be delivered to the colon intact. Upon reaching the colon, bacterial azo reductases cleave the compound to release 5-aminosalicylic acid, the therapeutically active portion of the molecule, and 4-aminobenzoyl-β-alanine. Balsalazide is
not official in any pharmacopoeia. Literature survey revealed several spectrophotometric methods for its quantitative estimation in bulk drug and pharmaceutical dosage forms.\[6-12\] But there is no method is reported, so in this work we attempt to develop a UV Spectrophotometric method for quantitative estimation of balsalazide formulation.

![Figure 1: Structure of Balsalazide.](image)

**MATERIAL AND METHODS**

**Apparatus**

Shimadzu UV-visible spectrophotometer (Model 2450) with 1 cm matched quartz cell was used in present study for spectral and absorbance measurements.

**Reagents and Materials**

All chemicals and reagents used were of analytical grade and double distilled water was used throughout the investigation.

- 0.1N NaOH: It was prepared according to I.P.1996
- Standard Stock Solution: Accurately weighed (100 mg) pure drug sample of balsalazide was transferred to 100 ml (1000 µg/ml) calibrated volumetric flask, dissolved and made up to the mark with 0.1N NaOH solution.

**Developed Method**

(1) Scanning for determination of maximum absorbance for pure drug in 0.1 N NaOH solution and preparation of calibration curve. By using the stock solution of 1000 µg/ml, transferred 10 ml into 100 ml volumetric flask and made up the volume (200 µg/ml) and subsequently dilution was carried out by withdrawing different aliquots (0.5, 1.0, 2.0, 4.0, 5.0 ml) from standard solution were transferred into a series of 10 ml calibrated flasks and all were made up to the mark with 0.1 N NaOH solution and absorbance was measured at 454 nm against blank. A calibration curve was plotted from the absorbance values so obtained. A
representative spectra and calibration curve of balsalazide is reported in Figure 2 & 3 respectively.

Figure 2: Spectra of Balsalazide (pure drug) in 0.1 N NaOH.

Figure 3: Calibration curve of Balsalazide.

Figure 4: Overlay Spectra of Balsalazide (pure drug) in 0.1 N 0.1 N NaOH.
Analysis of capsule Formulation
Twenty capsules were weighed accurately the powder equivalent to 10 mg of balsalazide was weighed and dissolved in about 75 ml 0.1 N NaOH. The solution was shaken thoroughly for about 15-20 min, and filtered using Whatman No. 41 filter paper; residue was washed with 20 ml 0.1 N NaOH. Filtrate and washing were transferred to a 100 ml calibrated volumetric flask and 0.1 N NaOH was added up to the mark (200 μg/ml). The 4 ml of above filtrate was diluted to 100 ml with 0.1 N NaOH. Absorbance was measured at 454 nm wavelength maxima and the concentration of the drug in sample solution was determined from calibration curve. The results of analysis are presented in Table 2 respectively.

Accuracy (recovery test): Accuracy of the method was studied by recovery experiments. The recovery experiments were performed by adding known amounts of the drugs in powdered capsules. The recovery was performed at three levels, 60, 80 and 100% of balsalazide standard concentration. The recovery samples were prepared in aforementioned procedure. Three samples were prepared for each recovery level. The solutions were then analyzed and the percentage recoveries were calculated from the calibration curve.

Table 1: Quantitative parameters of spectrophotometric method.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\lambda_{\text{max}}$/ nm</td>
<td>454</td>
</tr>
<tr>
<td>Beer’s law limits, μg/ml</td>
<td>10-100</td>
</tr>
<tr>
<td>Regression equation</td>
<td>$y = 0.015x - 0.015$</td>
</tr>
<tr>
<td>Slope</td>
<td>0.015</td>
</tr>
<tr>
<td>Intercept</td>
<td>0.015</td>
</tr>
<tr>
<td>Correlation coefficient ($r^2$)</td>
<td>0.998</td>
</tr>
</tbody>
</table>

Table 2: Results of Analysis of Capsule Formulation and Recovery Studies.

<table>
<thead>
<tr>
<th>Brand</th>
<th>Label claim</th>
<th>% Label claim Estimated*</th>
<th>Standard Deviation</th>
<th>% Recovery**</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cozabal</td>
<td>750μg/ml</td>
<td>99.45</td>
<td>0.240</td>
<td>98.98</td>
</tr>
<tr>
<td>Balacol</td>
<td>750μg/ml</td>
<td>99.17</td>
<td>0.180</td>
<td>99.12</td>
</tr>
</tbody>
</table>

*Average of six determinations

** Average of Recovery Studies at three different concentration levels
RESULTS AND DISCUSSION

UV method has been developed for the quantitative estimation of balsalazide from capsule formulation. The developed method is based on the solubility of balsalazide in 0.1N NaOH. The results of analysis from capsule formulation were within the permissible limits and the results of recovery studies reflect nil interference from excipients. The developed method was found to be simple, accurate and economical hence can be used for routine analysis of balsalazide from pharmaceuticals.

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REFERENCES


