VALIDATION OF UV SPECTROSCOPIC METHOD OF ANALYSIS FOR ASSAY AND DISSOLUTION OF ACECLOFENAC TABLET

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ABSTRACT
Aceclofenac is a non steroidal anti-inflammatory drug with good analgesic and anti-rheumatic properties. Various methods for analysis of the same are available but are time consuming and expensive due to solvents used. Here I have developed a new, precise and simple UV spectrophotometric method for estimation of aceclofenac from tablet formulation. The drug obeyed the Beer’s law and showed good correlation. It showed absorption maxima at 276 nm; in methanol for assay and Phosphate buffer pH 6.8 for dissolution. The linearity was observed between 0-120 mcg/ml. The results of analysis were validated by recovery studies. The recovery was more than 99%. The method was found to be very simple. Accurate, precise, economical and robust.

Keywords: Aceclofenac, UV-Vis spectrophotometry Recovery.

INTRODUCTION
Aceclofenac is a not steroidal anti-inflammatory drug with good analgesic and anti-rheumatic properties. Chemically it is [[2-[(6-Dichlorophenyl) amino] phenyl] oxy] acetic acid. It is used in various pain conditions like rheumatoid arthritis, osteoarthritis and ankylosing spondylitis.\(^1\),\(^2\),\(^3\),\(^4\)

It is official in British Pharmacopoeia.\(^4\) Several analytical techniques like titrimetric\(^4\),\(^5\), colourimetric\(^7\), spectroflurimetric\(^7\), densitometric\(^8\),\(^9\), HPLC\(^10\)-\(^11\), RP-hplc\(^12\),\(^13\), spectrophotometric\(^14\),\(^15\) and stripping voltammetric\(^16\) have been reported for assay of Aceclofenac. However some of these methods are costlier and time consuming. To
overcome these difficulties spectrophotometric analysis serves to be the quick, promising and reliable method for routine quality control analytical use.

The aim of the present study is to develop a new simple, rapid, reliable and precise UV spectrophotometric method for analysis of aceclofenac from tablet formulation; method is based on measurement of UV absorbance of aceclofenac in methanol and phosphate buffer pH 6.8.

MATERIALS AND METHODS

Instrument Used
A. Shimadzu UV-Vis Spectrophotometer Model Shimadzu 1700 and UV-Vis Spectrophotometer model Systronics 119 with
- Spectral bandwidth of 1mm, Wavelength accuracy 0.1mm
B. Bath Sonicator

Reagents and Solution
All the reagents used in this assay were of analytical grade and the reagent solutions were prepared using glass double distilled water. Aceclofenac pure drug was obtained as a gift sample from Shelly’s pharmaceutical. Aceclofenac Tablets were purchased from local market and experimental formulation for analysis. The methanol was used as a solvent for the assay and Phosphate buffer pH 6.8 for dissolution test medium.

EXPERIMENTAL

Determination of \( \lambda_{max} \):

![Fig.1: UV Scan of Aceclofenac in methanol](image-url)
Weighed amount of Aceclofenac was dissolved in Methanol to obtain a 0.1mg/mL solution. This solution was subjected to scanning between 200-400nm and absorption maximum was determined. No effect of dilution on absorption maxima was detected.

**Standard Stock Solution:**
A stock solution containing 1000mcg/mL of pure drug was prepared by dissolving 50mg of Aceclofenac in sufficient methanol to produce 50mL solution in a volumetric flask.

**Table No.1:** Optical characteristics and precision

<p>| | |</p>
<table>
<thead>
<tr>
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<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Absorption maxima</td>
<td>276mm</td>
</tr>
<tr>
<td>Beer’s law limit</td>
<td>0-120mcg/mL</td>
</tr>
<tr>
<td>Coefficient of Correlation</td>
<td>0.9997</td>
</tr>
<tr>
<td>Regression equation</td>
<td>Y=0.003x+0.006</td>
</tr>
<tr>
<td>Slope</td>
<td>0.0036</td>
</tr>
<tr>
<td>y intercept</td>
<td>0.0062</td>
</tr>
</tbody>
</table>

**Working standard solution**
10mL of the stock solution was further diluted to 100mL with methanol to obtain a working standard solution containing 100mcg/mL

**Linearity and Calibration**
The aliquots working standard solution was diluted serially with sufficient methanol to obtain the concentration range of 80-120 mcg/mL. A calibration curve for aceclofenac was obtained by measuring the absorbance at the $\lambda_{max}$ of 276 nm. Statistical parameters like the slope, intercept, coefficient of correlation, standard deviation, Relative standard deviation, and error were determined.

**Assay**
Accurately weighed the 20 tablets and powdered. The powder equivalent to 100mg of aceclofenac was transferred to 100mL volumetric flask and made the volume to mark with methanol this mixture and filtered through Whatmann filter paper No.41. Transferred 5mL of the filtrate into a 50mL. Volumetric flask and made the volume to mark with methanol. Determined the respective absorbance at 276mm against the methanol as blank. Two
commercially different formulations of manufacturers were used for study namely local Shelys Pharmaceuticals Limited and imported Intas Limited.

Recovery studies
Recovery studies were performed to judge the accuracy of the method. The study was performed in the range of 80% to 120% recovery of the labelled claim (100mcg/mL). The respective absorbance at 276 nm was recorded against the blank. The amount of added concentration was determined from the absorbance values obtained and percent recovery was determined for each formulation.

**Fig 2: Calibration curve of Aceclofenac in methanol**

<table>
<thead>
<tr>
<th>CONCENTRATION</th>
<th>ABS</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.008</td>
<td>0.281</td>
</tr>
<tr>
<td>0.009</td>
<td>0.315</td>
</tr>
<tr>
<td>0.01</td>
<td>0.351</td>
</tr>
<tr>
<td>0.011</td>
<td>0.389</td>
</tr>
<tr>
<td>0.012</td>
<td>0.423</td>
</tr>
</tbody>
</table>

Robustness
The evaluation of robustness was performed for system suitability to ensure the validity of analytical procedure. This was done by varying the analyst, time of study and instrument. The analysis was performed on UV-Vis. Spectrophotometer model Shimadzu 1700 and UV-
Vis Spectrophotometer model Systronics 119. Interday and Intraday analysis was performed by changing the analyst.

RESULTS

The UV scan of standard solution between 200-400nm showed the absorption maxima at 276nm, shown in Fig. 1. The Beer’s law was verified from the calibration curve by plotting a graph of concentration vs. absorbance. The plot is shown in fig. 2.

Regression analysis showed very good correlation. The calibration plot revealed zero intercept which is clear by the regression analysis equation $Y = mX + C$ (where $Y$ is absorbance, $m$ is the slope and $X$ is the concentration of aceclofenac in mcg/mL) as obtained by the least square method. The results of analysis for assay and recovery studies for three different formulations were studied and are shown in Table No.II.

No significant variations were observed on interday and intraday analysis. Also no significant variations were observed on changing the instrument make and model.

Table No. II: Results of analysis

<table>
<thead>
<tr>
<th>Formulation</th>
<th>Label Claim</th>
<th>%Label Claims found</th>
<th>Standard Deviation</th>
<th>Coefficient Of Variation</th>
<th>Standard Error</th>
<th>% Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>DC001</td>
<td>100 mg</td>
<td>99.801</td>
<td>0.2905</td>
<td>0.2675</td>
<td>0.1477</td>
<td>99.8</td>
</tr>
<tr>
<td>DC002</td>
<td>100 mg</td>
<td>99.666</td>
<td>0.2565</td>
<td>0.2341</td>
<td>0.1381</td>
<td>99.7</td>
</tr>
<tr>
<td>DC003</td>
<td>100 mg</td>
<td>100.925</td>
<td>0.2565</td>
<td>0.2441</td>
<td>0.1481</td>
<td>99.6</td>
</tr>
</tbody>
</table>

DISCUSSION

The spectrum of aceclofenac in methanol showed the absorption maxima at 276nm. No effect of dilution was observed on the maxima, which confirmed the maxima at 276 nm. The statistical analysis of data obtained for the calibration curve of aceclofenac in pure solution indicated a high level of precision for the proposed Method, as evidenced by low value of coefficient of variation. The coefficient of correlation was highly significant. The linearity
range was observed between 0 – 120mcg/mL. The plot clearly showed a straight line passing through origin (Y = 0.036 X + 0.062).

The assay method was validated by low values of % RSD and standard error, indicating accuracy and precision of the methods. Excellent recovery studies further proves the accuracy of the method.

Robustness of the method was studied by varying the instrument, time of study and analyst. Reproducibility of the results confirmed the robustness of the method.

CONCLUSIONS
From the results and discussion the method described in this paper for the determination of aceclofenac from tablet formulation is simple, accurate, sensitive and reproducible. The proposed method utilizes inexpensive Solvents. The proposed method could be applied for Routine quality analysis in quality control laboratories of pharmaceutical formulation companies.

ACKNOWLEDGEMENTS
Author is grateful to Shelys Pharmaceuticals Limited, Dar-es-Slalaam for providing the gift sample of Aceclofenac. We are also Thankful to the Dean ,School of Pharmacy ,Muhimbili University of Health and Allied Sciences ,Dar- es-Salaam for providing the necessary facilities to carry out this work.

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