Stability studies for the determination of shelf life of aceclofenac formulation

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\textbf{ABSTRACT}

Stability studies for optimized formulations were carried out according to ICH guidelines. The optimized formulations were subjected to accelerated stability studies \cite{1}. Sufficient replicates of formulation were prepared, packed in aluminium foil and stored in petri dishes at temperature of 40± 0.5°C, 50± 0.5°C and 60± 0.5°C for 60 days. Samples were withdrawn at intervals of 15, 45 and 60 days and analyzed for drug content by HPLC method. The shelf life of formulations of aceclofenac were determined by accelerated stability studies on the basis of first order degradation kinetics and $t_{0.9}$ (the time required to degrade 10\% of drug at 25°C). The shelf life was found to be 1.469 yrs.

\textbf{Keywords:} Aceclofenac, Formulation, Stability studies, Shelf life.

\textbf{MATERIALS AND METHODS}

The HPLC method was performed according to Ph Eur monograph 1281 \cite{2}, with slight modification as per our system availability. The system consisted of Thermo finagnn with a UV detector (Model: Surveyor autosampler plus). In this method Acetonitrile: Water (9:1): Phosphoric acid (70:30) optimized as a mobile phase plus diluent and a 10 cm X 4.6 mm RP C\textsubscript{18} Hypersil gold column having a 5µm packing as a stationary phase. Flow rate of 1.0 ml/min, Detection at 275 nm, Injection volume of 10 µl was used.

Ten different concentrations of aceclofenac ranging from 2– 20 µg/ml were prepared for linearity studies (Table-1). The responses were measured as peak areas and plotted against concentrations to prepare a calibration curve (Fig-1).

\begin{table}[h]
\centering
\begin{tabular}{|c|c|c|}
\hline
Concentration (µg/mL) & Area & S.D. (n=3) \\
\hline
2 & 52000 & ± 37 \\
4 & 100000 & ± 125 \\
6 & 180000 & ± 306 \\
8 & 280000 & ± 1105 \\
10 & 380000 & ± 1285 \\
12 & 460000 & ± 1956 \\
14 & 570000 & ± 2384 \\
16 & 699443 & ± 5695 \\
18 & 810000 & ± 6820 \\
20 & 900000 & ± 10785 \\
\hline
\end{tabular}
\caption{Concentration and the Area obtained for construction of calibration curve.}
\end{table}
**Table 2: Degradation of aceclofenac Formulation at different temperatures**

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Drug Content (mg)</th>
<th>% Drug Remaining</th>
<th>Log % Drug Remaining</th>
<th>Drug Content (mg)</th>
<th>% Drug Remaining</th>
<th>Log % Drug Remaining</th>
<th>Drug Content (mg)</th>
<th>% Drug Remaining</th>
<th>Log % Drug Remaining</th>
</tr>
</thead>
<tbody>
<tr>
<td>40 ± 0.5°C</td>
<td>49.7</td>
<td>100</td>
<td>2</td>
<td>49.8</td>
<td>100</td>
<td>2</td>
<td>49.7</td>
<td>100</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>49.6</td>
<td>99.79</td>
<td>1.999087</td>
<td>49.5</td>
<td>99.39</td>
<td>1.997343</td>
<td>49.21</td>
<td>99.01</td>
<td>1.995679</td>
</tr>
<tr>
<td></td>
<td>49.4</td>
<td>99.39</td>
<td>1.997343</td>
<td>49.1</td>
<td>98.59</td>
<td>1.993833</td>
<td>48.83</td>
<td>98.24</td>
<td>1.992288</td>
</tr>
<tr>
<td></td>
<td>48.9</td>
<td>98.39</td>
<td>1.992951</td>
<td>48.92</td>
<td>98.23</td>
<td>1.992244</td>
<td>48.56</td>
<td>97.70</td>
<td>1.989895</td>
</tr>
</tbody>
</table>
The logarithm of % drug remaining was plotted against time in days (Fig.-2), which gave almost straight line suggesting that drug degradation followed first order kinetics. The slope of the straight line for each temperature was obtained and the degradation rate constant was calculated using the formula given below:

\[
\text{Slope} = \frac{-K}{2.303}
\]

Where, K is degradation rate constant.

An Arrhenius plot was drawn by plotting logarithm of K values against reciprocals of absolute temperature (Fig.-3). The value of K at 25°C (K_{25}) was extrapolated from the Arrhenius plot and shelf-life of the formulation was calculated by substituting the value of K_{25} in the following equation:

\[
t_{0.9} = 0.1054/K_{25}
\]
Where, t₀.₉ is the time required for 10% degradation of the drug and is referred to as the “Shelf-life” of the product. The degradation rate constant at various temperatures and the shelf life of the formulation is reported in Tables-2.

![Arrhenius plot for optimized aceclofenac](image)

**Fig.-3: Arrhenius plot for optimized aceclofenac**

**CONCLUSION**

In this study, the shelf life of transdermal formulations of Aceclofenac was found to be 1.469 yrs.

**REFERENCES**