A Review: Analytical Methods For Determination Of Diclofenac In Pharmaceutical Samples

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Abstract: Diclofenac is NSAID’S drugs which is available in different dosage form like Tablet, Capsule, Syrup, Cream. This article reviews the analytical methods for identification and quantitative determination of Diclofenac in sample. The most commonly adapted method for determination of Diclofenac in sample on UV- Spectrometry (Spectrofluorometry, Colorimetry), Chromatography like High profile liquid chromatography (HPLC), Gas chromatography with mass spectrometry and High performance thin layer chromatography (HPTLC).

Keywords: Diclofenac, Ibuprofen, colorimetric, HPLC, HPTLC and Gas chromatography

I. INTRODUCTION

Non steroidal antiinflamentry drugs (NSAID’S) have analgesic, antiinflamentry, antipyretic properties. The NSAID’S act by blocking of enzyme cycloxygenase i.e. COX and hence there is inhibition of formation of prostaglandin from arachidonic acid which is part of phospholipids. NSAID’S have small or no effect on lipoygenase which are convert leukotriene from arachidonic acid. Both prostaglandin and leukotriene have effect in inflammation process, NSAID’S inhibit synthesis of prostaglandins within central nervous system which exert antinociceptine action and it also act by blocking platelet cycloxygenase, which inhibit formation of thromboxane A2 known as aggregating agent e.g. Aspirin.

II. CLASSIFICATION OF NSAID’S DRUG

The classification NSAID’S based upon clinical pharmacological characters, half life, chemical classification.
✓ Salicylic acid derivatives: Aspirin, Sodium salicylate, Olsalazine, Diflunisal, Salicylsalicylic acid and Sulfasalazine
✓ Para-aminophenol derivatives: Acetaminophen
✓ Pyrazolone derivatives: Metamizol
✓ Non steroidal anti-inflammatory drugs-
  • Indoleacetic acid: Indomethacin, etodolac, and Zomepirac
  • Benzothiazide or Oxicam derivatives: Tenoxicam, Piroxicam and Meloxicam
  • Pyrrole acetic acid derivatives: Alclofenac, Diclofenac, Bromfenac and Ketorolac
  • Propionic acid derivatives: Ibuprofen, fenoprofen, Ketoprofen and Suprofen
  • Arylalkanoic acid derivatives: Nabumetone
  • Benzothiazide or oxicam derivatives: Piroxicam, Meloxicam and Meloxicam
  • COX-2 selective inhibitors: Rofecoxib, Celecoxib and Nimesulide
✓ Gold compound:Auranofin, Gold sodium thiomalate
✓ Antigout drugs: Colchicine, Probancid, Sulfinpyrazone

III. ADVERSE EFFECT OF NSAID’S

CNS- Headache, Vertigo, Dizziness, Hyperventilation, Confusion
CVS-Myocardial infarction, Closure of ductusarteriosus Hypersensitivity- Asthma, Shock, Urticaria, Hypotension, Flushing
Platelets-Increased risk of haemorrhage, Inhibited platelet activation
GI-Nausea, Anorexia, Abdominal pain, Ulcer, Diarrhoea
Renal- Hyperkalemia, Salt and retention, Decreased urate excretion, Decreased effectiveness of diuretic medications
Uterus-Inhibition of labour, Prolongation of gestation

IV. DICLOFENAC

The Diclofenac is Aryl acetic derivatives. The chemical name of Diclofenac is 2-[2,6-dichlorophenylamino] benzene acetic acid. It is white to slightly yellowish crystalline powder, sparingly soluble in water, freely soluble in ethanol, methanol. It is An analgesic, antipyretic, antiinflammatory drug, which act by inhibition of prostaglandin synthesis by selecting COX-2.

A. PHARMACOKINETICS

Diclofenac is mostly absorbed orally. It is protein bound 99% metabolized and excreted in urine and bile. It is also found in synovial fluid is maintained for 3 times longer period than in plasma.

B. USES

Diclofenac is used in rheumatoid, osteoarthritis and dysmenorrhea, bursitis, relief pain and wound oedema. It is used in treatment of spondylitis.

C. DOSE

50 mg, 100mg of enteric coated tab. And S.R. tab. respectively with brand name VOVERAN, DICLONAC, MOVONAC and 25 mg/ml in 3ml amp. For i.m. inj.

V. METHODS FOR DETERMINATION OF DICLOFENAC

- Spectrometry
- Spectrofluorometry
- Colorimetry
- Chromatography

A. SPECTROMETRY

<table>
<thead>
<tr>
<th>Wavelength</th>
<th>Sample Matrix</th>
<th>Solvent</th>
<th>Linearity</th>
<th>Accuracy</th>
<th>Precession</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>246-260 nm</td>
<td>Paracetamol, Diclofenac</td>
<td>Methanol</td>
<td>0.9999</td>
<td>99.81-100.2</td>
<td>0.0979</td>
<td>[6]</td>
</tr>
<tr>
<td>276 nm</td>
<td>Potassium, Thiochococyste</td>
<td>Methanol</td>
<td>7.5</td>
<td>100.14-101.98</td>
<td>0.01-1</td>
<td>[7]</td>
</tr>
<tr>
<td>252-277 nm</td>
<td>Paracetamol, Potassium, Diclofenac</td>
<td>Methanol/Diis water(40:60)</td>
<td>2-30</td>
<td>98.22-102.76</td>
<td>1.42</td>
<td>[8]</td>
</tr>
<tr>
<td>277-283 nm</td>
<td>Paracetamol, Potassium, Methanol, Diclofenac</td>
<td>Methanol</td>
<td>0.9992</td>
<td>97.017-98.339</td>
<td>0.4801-1.472</td>
<td>[9]</td>
</tr>
<tr>
<td>252-277 nm</td>
<td>Paracetamol, Methanol, Diclofenac</td>
<td>Methanol</td>
<td>0.9999</td>
<td>98.26-101.16</td>
<td>0.6255</td>
<td>[10]</td>
</tr>
<tr>
<td>276 nm</td>
<td>Urea, Diclofenac</td>
<td>Methanol</td>
<td>0.9991</td>
<td>96.6-0.613</td>
<td>0.177-0.361</td>
<td>[11]</td>
</tr>
<tr>
<td>249 nm</td>
<td>Diclofenac</td>
<td>Methanol</td>
<td>4-36</td>
<td>99.02-99.46</td>
<td>0.76-1.81</td>
<td>[12]</td>
</tr>
<tr>
<td>566.2 nm</td>
<td>Diclofenac, Thiochococyste</td>
<td>Toluene</td>
<td>0.6-10</td>
<td>82.6-94.3</td>
<td></td>
<td>[13]</td>
</tr>
</tbody>
</table>

Table 1: Condition for UV-Spectrometry analysis for Diclofenac in samples

B. SPECTROFLUOROMETRIC METHOD

Spectrofluorometric method of determination of Diclofenac Sodium in pharmaceutical tablet and ointment using Shimadzu RF-5301 PC. Spectrofluorometer equipped with a 150W Xenon arc Lamp, using 1.00cm quartz cells. The fluorescence intensity of Diclofenac in acid solution (HCl 0.01 M) It exciting at 289 nm and obtaining fluorescence emission at 562 nm.

C. COLORIMETRIC METHOD

A simple and precise colorimetric method was developed for determination of Diclofenac Sodium in tablets dosage form, using newly developed 4-Carboxyl-2,6-dinitrobenzediazoniation (CDNBD) as chromatographic derivatizing reagent with azo dye. Diclofenac exhibit absorbance at 470 nm and obeyed linearity in concentration range 1.35-10.8 g/ml. The LOQ and LOD were found to be 0.81 and 0.27 g/ml respectively. This method has advent of speed, simplicity, sensitivity and affordable instrumentation.

D. CHROMATOGRAPHY

a. HPLC

<table>
<thead>
<tr>
<th>Detector</th>
<th>Samples Matrix</th>
<th>Chromatographic column</th>
<th>Mobile phase</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>UV at 243 nm</td>
<td>Diclofenac, Benzoic acid</td>
<td>Zebron C-18</td>
<td>0.1%Glacial acetic acid and Water Acetonitrile:58.5 (v/v)</td>
<td>[16]</td>
</tr>
<tr>
<td>Electrochemical detector</td>
<td>Diclofenac</td>
<td>C18(250mmx4.6mm)</td>
<td>Methanol: Water(90:10)</td>
<td>[16]</td>
</tr>
<tr>
<td>UV at 240 nm</td>
<td>Paracetamol, NSAID</td>
<td>WATERS: XTERRA</td>
<td>Methanol: Water(90:10)</td>
<td>[17]</td>
</tr>
<tr>
<td>UV Visible</td>
<td>Diclofenac</td>
<td>RP-H8(6150)5</td>
<td>Methanol:0.5%Phosphoric acid</td>
<td>[19]</td>
</tr>
</tbody>
</table>

Table 2: Condition for HPLC analysis for Diclofenac in sample
b. **HPTLC**

<table>
<thead>
<tr>
<th>Scanning/ Detector</th>
<th>Samples Matrix</th>
<th>Stationary phase</th>
<th>Mobile phase</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Camag TLC</td>
<td>Diclofenac Sodium, Fumoteline</td>
<td>Aluminum 200 layer of Silica gel 60 RP 18</td>
<td>Methanol: Water: Triethylamine (7:5.3:5.0:5)</td>
<td>[20]</td>
</tr>
<tr>
<td>Camag TLC</td>
<td>Diclofenac HCL&amp; Tolperisone</td>
<td>Silica gel 60F254</td>
<td>Tolune: Ethyl acetate: Methanol (4:4:2 ml v/v)</td>
<td>[21]</td>
</tr>
<tr>
<td>Camag TLC</td>
<td>Diclofenac Sodium, Tolune:Ethyl acetate: Methanol (5:3.2:2 v/v)</td>
<td>[22]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Camag TLC</td>
<td>Diclofenac Potassium, Chlorzoxazone</td>
<td>Silica gel 60F254</td>
<td>Tolune Ethyl acetate (5:45 v/v)</td>
<td>[23]</td>
</tr>
<tr>
<td>Scanning-3 TLC</td>
<td>Tramadol.</td>
<td>Merck TLC</td>
<td>Chloroform: Tolune (4:2:2,6 v/v/v)</td>
<td>[24]</td>
</tr>
<tr>
<td>Scanning-3 TLC</td>
<td>Diclofenac</td>
<td>Silica gel 60F254</td>
<td>Methanol:Ethyl acetate: Triethylamine (7:5.3:5.0:5)</td>
<td>[24]</td>
</tr>
</tbody>
</table>

**Table 3: Condition for HPTLC analysis for Diclofenac in sample**

**c. **G**AS CHO**R**MATOGRAPHY**

The rapid, sensitive and specific methods were developed for determination of Diclofenac in pharmaceutical preparation by gas chromatography with mass spectrometry. The linearity was established over concentration range 0.25-5g/mL. The intra- and inter-day relative standard deviation (RSD) was less than 4.62%. The limits of quantification (LOQ) were determined as 0.15g/mL. This method is used for quality control of Diclofenac pharmaceutical dosage form to quantify drug and check formulation content uniformity.

VI. CONCLUSION

The presented systematic review discuss about various analytical method for the determination of Diclofenac in pharmaceutical dosage form samples. These analytical methods are important for qualitative and quantitative determination of Diclofenac in pharmaceutical dosage form.

**REFERENCES**


