Abstract
Simultaneous Determination of Sildenafil Citrate and Dapoxetine Hydrochloride in Pharmaceutical Formulation by HPLC Utilizing Design of Experiments (DoE)

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Recently, Sildenafil citrate (SILD) has been co-formulated with Dapoxetine hydrochloride (DAPO) as combined tablet formulations which are used for treatment of male sexual disorders as erectile dysfunction (ED) and premature ejaculation (PE).

In this work, a new, reliable, economic and reproducible method of High Performance Liquid Chromatography with Photo-Diode Array detection (HPLC-PDA) was developed for simultaneous determination of SILD and DAPO in raw material and marketed formulation.

This method was optimized using an approach of design of experiments (DoE) using central composite design (CCD) after studying the critical analytical attributes and independent factors. The optimized mobile phase was consisted of methanol, acetonitrile and ammonium acetate buffer (containing 0.025% triethyl amine) in ratio of 32.26:42 (v/v/v). The final pH of mobile phase was adjusted to 5.10 ± 0.10 using glacial acetic acid.

Quantitative and qualitative analyses were carried on Hypersil - Gold C18 column (4.6 mm i.d. x 150 mm, 5.0 μm particle size, Thermo-Fisher, USA) and detection at wavelength of 230 nm. This analytical method was validated in accordance with The International Council for Harmonisation of Technical Requirements for Pharmaceuticals (ICH) guidelines as system suitability tests; linearity, range, accuracy, precision, limit of detection (LOD), limit of quantitation (LOQ) and robustness.

The linearity range of the method for SILD and DAPO was ranged from 1.25 to 50 μg/ml and 0.75 to 50 μg/ml respectively. LOD of SILD and DAPO were 0.0121 μg/ml and 0.0112 μg/ml respectively, while LOQ of SILD and DAPO were 0.0366 μg/ml and 0.0339 μg/ml respectively. The precision of this chromatographic method was less than 2% for repeatability, intra-day precision and inter-day precision. The accuracy of the method was ranged from 99.1 - 101.75 %.

Degradation tests were carried out to study the effect of stress conditions (acidic, alkaline, thermal, oxidation and photo - stability) on active pharmaceutical ingredients (APIs) and tablet dosage form. The dissolution tests were conducted to calculate the percent drug dissolved from the marketed dosage form.

In conclusion, the optimized method is sensitive, rapid, accurate, precise, economic, robust, rugged and selective for the routine analysis of pharmaceutical formulations (tablet) and raw material of sildenafil and dapoxetine in quality control labs.

Detailed view of the monograph

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