Stability Indicating HPLC Method for determination of Tadalafil in Raw Material and Pharmaceutical Formulation using Design of Experiments (DoE)

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Abstract
The purpose of this study was to develop a new high performance liquid chromatographic method for determination of tadalafil in tablet dosage form. In the present study a rapid, simple, economical HPLC-PDA procedure has been developed and validated.

The HPLC method was developed and optimised using Central-composite-design (CCD), after studying the physicochemical properties of tadalafil (solubility, melting point, IR, UV). The Optimised mobile phase was [55% buffer (25 mM Na₂HPO₄), 45% acetonitrile and the PH value was adjusted to 7.00 ±0.05], using orthophosphoric acid 85%. The flow rate was 1.0 ml/min, signals were detected at wave length of 267nm.

The linearity range of the method was from 2 to 24 μg/ml for tadalafil. LOD was 0.0789 μg/ml for tadalafil, whereas LOQ was 0.239 μg/ml. The precision of the method was less than 2% which indicates highly precise result during the study. The accuracy of the method were ranged from 99.6%-102.79%.

In conclusion the method presented here is sensitive, rapid, accurate, precise, economic, robust, rugged and selective for the routine analysis of the drug in formulation (tablet) and raw material without changing the chromatographic parameters.

The second part of the work was to develop validate gradient method for separation of impurity (Trans tadalafil), and for studying the effect of stress conditions on raw material and tablet dosage form.

There was no degradation products of tadalafil after exposure to sun light, and sample exposed to stress condition (30°C,75%RH, 3months). Study showed that the products are degraded during stress conditions (after exposure to acidic, alkaline and hydrogen peroxide).

Detailed view of the monograph
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