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Research Article

QUANTITATIVE DETERMINATION OF SILDENAFIL CITRATE IN COMMERCIAL TABLET DOSAGE FORM MARKETED IN MAIDUGURI METROPOLITAN COUNCIL (MMC)

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ABSTRACT
A quantitative analysis was carried out to determine the claimed content of sildenafil citrate present in commercial tablet dosage forms of different brands, using the reference standard from the developed method for assay of sildenafil citrate. Nine different samples were analysed using HPLC and UV-Spectrophotometric method.
For the HPLC and UV Spectrophotometer result, Pramo V has a percentage content of 103.7% and 102.6%, power 97.5 and 96.5% Man- up 95.2% and 96.9%, soga 104.2% and 104.6, caverta 101.9% and 100.2%, Homograe 102.8% and 100.5% , Teagra 99.0% and 98.4%, vega 91.3% and 91.4% and ceagra 109.1% and 114.4% , respectively. It was observed that both vega and ceagra samples has a percentage concentration of 91.3, 91.4% and 109.1% , 114.4% respectively. They thus failed the analysis test as the percentage content did not fall within the standard range of 95%-105%
Based on the result obtained, about 78% of drugs that contain sildenafil citrate passed the analysis, while 22% failed.
Keywords: Sildenafil, HPLC, Ultra Violet Spectrophotometry

INTRODUCTION

Sildenafil citrate is a drug popularly marketed as Viagra by Pfizer. Is a potent and selective inhibitor of cyclic guanosine monophosphate (cGMP)-specific phosphodiesterase type V (PDE V), the predominant isozyme metabolizing cGMP in the corpus cavernosum[1] (Goldstein, 1998). Sildenafil citrate is chemically designated as 1-[[3-(6,7-dihydro- 1-methyl-7-oxo-3-propyl-1H-pyrazolo[4,3-d]pyrimidin-5-yl)-4-ethoxyphenyl] sulfonyl]-4-methylpiperazine citrate (Martindale). It is marketed as an oral agent to treat male erectile dysfunction. It is an ampholyte with pKa value 4 (pirydinium ion) and 8.8 (benzimidazole). Sildenafil citrate is twice more soluble in methanol than in water. Its solubility decreases with pH up to 9 when it starts to increase again

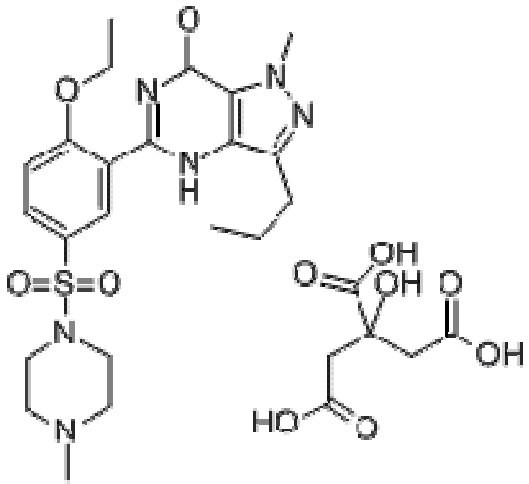
QUANTIFICATION OF SAMPLE USING UV SPECTROPHOTOMETRY

For perfect quantification of sample, it depends on the absorbance produced by the specific concentration of the sample and that of the standard.

Thus; Percentage of Sample__ Absorbance of sample × 100
Absorbance of Standard

Cooper et al. have developed a procedure for simultaneous determination of sildenafil and its metabolite in plasma using automated sequential trace enrichment of dialysates[2] a reversed phase HPLC methods have been utilized for the determination of sildenafil citrate in dosage forms[3]. Cooper et al. have developed a procedure for simultaneous determination of sildenafil and its metabolite in plasma using automated sequential trace enrichment of dialysates[2], a reversed phase HPLC methods have been utilized for the determination of sildenafil citrate in dosage forms[3]. Ashok k. also estimated the sildenafil citrate in bulk and in tablet dosage form[4]. All these quantitative analysis of sildenafil citrate where approved to be referenced in international journals as article to the public.

CHEMICAL STRUCTURE OF SILDENAFIL CITRATE[5]



http://www.chemicalbook.com/ChemicalProductProperty_EN_CB9407725.htm

GENERIC NAME, BRAND NAME AND OTHER TRADE NAMES

Generic name: sildenafil
Brand name: Viagra
Other trade names: caverta, Vega, ceagra, homograe, pramoV, teagra, and so on.

DRUG DESCRIPTION

Sildenafil Citrate is an off white crystalline powder. It is formulated as blue, film coated diamond shaped tablets. Tablets are manufactured at a dosage of 25mg, 50mg and 100mg.
Solubility: 3.5 mg/ml in water
Molecular weight: 666.7 g/mol

EXPERIMENTAL SECTION

METHOD FOR HPLC ANALYSIS
A High Performance Liquid Chromatograph system, with LC solutions data handling system

(HPLC UV-VIS, Perkin Elmer 2008) with an auto sampler was used for the analysis. The data was recorded using LaChrom 2008 solutions software. The determination performed on a stainless steel column 150 mm long, 4.6 mm internal diameter filled with Octadecyl silane chemically bonded to porous silica particles of 5 mm diameter (Inertsil C18, 5m, 150 mm x 4.6 mm, make: Perkin Elmer ltd, Japan) with the mobile phase containing acetonitrile and phosphate buffer in the ratio of 70:30 (v/v pH 7.0) at ambient temperature. Flow rate was kept at 1 ml/min, and the elution was monitored at 228 nm.

PREPARATION OF MOBILE PHASE:

Mobile phase was prepared by mixing 700 ml of acetonitrile with 300 ml of phosphate buffer and its pH adjusted to 7.0. The mobile phase was sonicated for 15 min and then it was filtered through a 0.45m membrane filter paper .

PREPARATION OF STOCK AND STANDARD SOLUTIONS:

The standard used was referenced from the developed method and it was sited below

Accurately weighed 25 mg of test sample into a clean dry 50 ml volumetric flask, dissolve and diluted the mark with mobile phase. Mark this solution as sample solution. This solution contains 0.5mg/ml of sample. Qualified working standard of Sildenafil Citrate is used to carry out validation exercise. The potency of working standard is 99.68 %. With the optimized chromatographic conditions, a steady baseline was recorded, the standard solution was injected and the chromatogram was recorded. This procedure was repeated for the sample solution[6]. For the reason of using this reference standard, the same as chromatographic condition was adopted.

SAMPLE PREPARATIONS

Nine different samples of tablet containing sildenafil citrate of different brands with expiry date not less than 365 days was used, five accurately weighed tablets were crushed to a fine powder and an amount equivalent to 10 mg of sildenafil citrate was added into different 100 ml volumetric flasks and volume was made up with the mobile phase. The samples were filtered through a 0.45-µm-membrane filter; a serial dilutions (50µg/ml) was made from this solution in 50 ml volumetric flask for each samples and were injected for HPLC analysis[6].

METHOD USED FOR UV-SPECTROPHOTOMETRIC ANALYSIS;

A lambda 35, UV-Visible double beam spectrophotometer with 1 cm matched quartz cell was used and nine different brands of tablets of sildenafil were obtained from local stores for the analysis.

SILDENAFIL STOCK SOLUTION:

Standard stock solution was prepared by dissolving 70.25 mg of Sildenafil in 100 mL of methanol to get concentration of 500 µg/mL solution[4].

PROCEDURE FOR CALIBRATION CURVE:

Aliquots of stock solution were further diluted with Methanol to get working solution of 5, 10, 15, 20, 25 and 30 µg ml - 1. Finally, the prepared standards were measured after standing for 5.0 min at λ max as recorded in each case against a solvent blank similarly prepared. A calibration graph of the absorbance versus the concentration of the drug was plotted[4]. For the reason of using this reference standard, the same spectrophometric condition was adopted.

PROCEDURE FOR THE ANALYSIS OF SAMPLE DOSAGE FORMS:

For analysis of commercial formulations, ten tablets were taken and powdered. Tablet powder equivalent to 140.5 mg of sildenafil citrate was transferred to 100 mL volumetric flask and dissolved in methanol. Then the solution was sonicated for 15 min and filtered and it was for further diluted to get the required concentration of 20ug/ml which is equivalent to 20ppm. The absorbance of the prepared sample solution was measure against methanol blank at 291±2 nm.

QUANTITATIVE JUSTIFICATION

The standard range used by both method of analysis for percentage content of tablet is 95-105%.

RESULTS AND DISCUSSION

Calculation of content of tablet on HPLC;

$$\text{Content of tablet (\%)} = \frac{\text{Peak Area of sample}}{\text{Peak Area of standard}} \times 100$$

Calculation of content of tablet on UV-Spectrophotometer;

$$\text{Content of tablet (\%)} = \frac{\text{Absorbance of sample}}{\text{Absorbance of standard}} \times 100$$

HPLC graphical result is shown bellow;

Table 1 showing information contained on the samples

Name of sample	Batch Number	Manufacturing date	Expiry date
Pramo V	B D001M	April,2010	March,2013
Power	VP02	April,2009	September,2011
Vega	S103	June,2009	May,2012
Man-up	T91016	October,2009	September,2012
Soga	GS 12	July, 2009	December,2011
Ceagra	T119209	November,2009	October,2012
Caverta	2150151	April,2010	March, 2012
Homogra	L1736	August,2009	July,2012
Teagra	UTT09	August,2008	July,2011

Table 2 showing percentage content of tablets in the samples using HPLC and UV-Spectrophotometry;

SAMPLES	HPLC %	UV-Spectrophotometry %
Pramo V	103.7	102.6
Power	97.5	96.5
Vega	91.3	91.4
Man-up	95.2	96.9
Soga	104.2	104.6
Ceagra	109.1	114.4
Caverta	101.9	100.2
Homograe	102.8	100.5
Teagra	99.0	98.4

DISCUSSION

In tablet production, uniformity of tablet quantity is very important so as to ascertain the specified quantity of active ingredient contained in the dosage form. Therefore, in quantitative analysis, percentage tablet content is determined to quantify the active ingredient being claimed by the label

From the result obtained, it was found that seven out of the analysed sample passed the test. For the HPLC and UV Spectrophotometer result, Pramo V has a percentage content of 103.7% and 102.6%, power 97.5 and 96.5% Man- up 95.2% and 96.9%, soga 104.2% and 104.6, caverta 101.9% and 100.2%, Homograe 102.8% and 100.5% , Teagra 99.0% and 98.4%, vega 91.3% and 91.4% and ceagra 109.1% and 114.4% , respectively. It was observed that both vega and ceagra samples has a percentage concentration of 91.3, 91.4% and 109.1% , 114.4% respectively. They thus failed the analysis test as the percentage content did not fall within the standard range of 95%-105%

CONCLUSION

Based on the result obtained, about 78% of drugs that contain sildenafil citrate passed the analysis, while 22% failed. Thus Vega sample was observed to contain 91.3 % and 91.4 % of 100mg of sildenafil citrate of label claims, and ceagra sample contain 109.1% and 114.4% of 100mg of sildenafil citrate of the label claim from the result of both HPLC and UV-Spectrophotometric method respectively. This studies thus emphasized on the quantitative determination of sildenafil citrate in commercial tablet dosage forms.

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