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STABILITY INDICATING RP-HPLC METHOD DEVELOPMENT AND VALIDATION FOR SIMULTANEOUS ESTIMATION OF SILODOSIN & TADALAFIL IN CAPSULE DOSAGE FORM

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ABSTACT

A simple, precise, accurate, economical and rapid stability-indicating reverse-phase high-performance liquid chromatographic (RP-HPLC) method has been developed and validated for the simultaneous estimation of Silodosin and Tadalafil in capsule dosage form. Chromatographic separation was achieved using a Aglilent zorbax C_{18} (250 mm x 4.6 mm x 5 µ) with a isocratic mobile phase consisting of acetate buffer (pH 3.7), acetonitrile and Trifluoracetic acid at a flow rate of 1 mL/min. Detection was carried out at 284 nm, with column temperature maintained at 25°C. Retention times were noted as 16.328 min for Silodosin and 11.255 min for Tadalafil respectively. Various analytical validation parameters, including specificity, linearity, LOD, LOQ, precision, accuracy, and robustness, were determined per ICH Q2 (R2) guidelines. Linearity was established over 80–240 µg/mL for Silodosin and 50–150 µg/mL for Tadalafil. The proposed method was successfully applied for the simultaneous estimation of both the drugs in commercial combined dosage form.

KEYWORDS: Silodosin, Tadalafil, RP-HPLC, estimation, forced degradation, validation, method, development.

INTRODUCTION

Benign prostatic hyperplasia (BPH) involves the non-cancerous enlargement of prostate tissue and ranks as a leading cause of lower urinary tract symptoms (LUTS) among aging men. Its occurrence rises steadily with age.[1]

Key features encompass bladder outlet obstruction, LUTS and benign prostatic enlargement (BPE). BPH specifically denotes the cellular changes in prostate tissue, while BPE indicates glandular enlargement (often linked to BPH) and bladder outlet obstruction refers to impaired urine flow. Patients with BPE experiencing such obstruction are classified as having benign prostatic obstruction.[1]

LUTS capture the urinary issues common to conditions impacting the prostate and bladder, most often driven by BPH. These descriptors have mostly supplanted the older term "prostatism."[1]

Silodosin[2,3]

It works as adrenergic alpha-Antagonists. Silodosin exerts its effect by acting on afferent nerves in the bladder, thereby reducing bladder over activity and alleviating storage.

Tadalafil[4,5]

It works as Phosphodiesterase 5 Inhibitors. Tadalafil is a selective inhibitor of phosphodiesterase-5 (PDE5) that triggers a cascade of downstream effects, the most prominent therapeutic outcome being relaxation of smooth muscle.

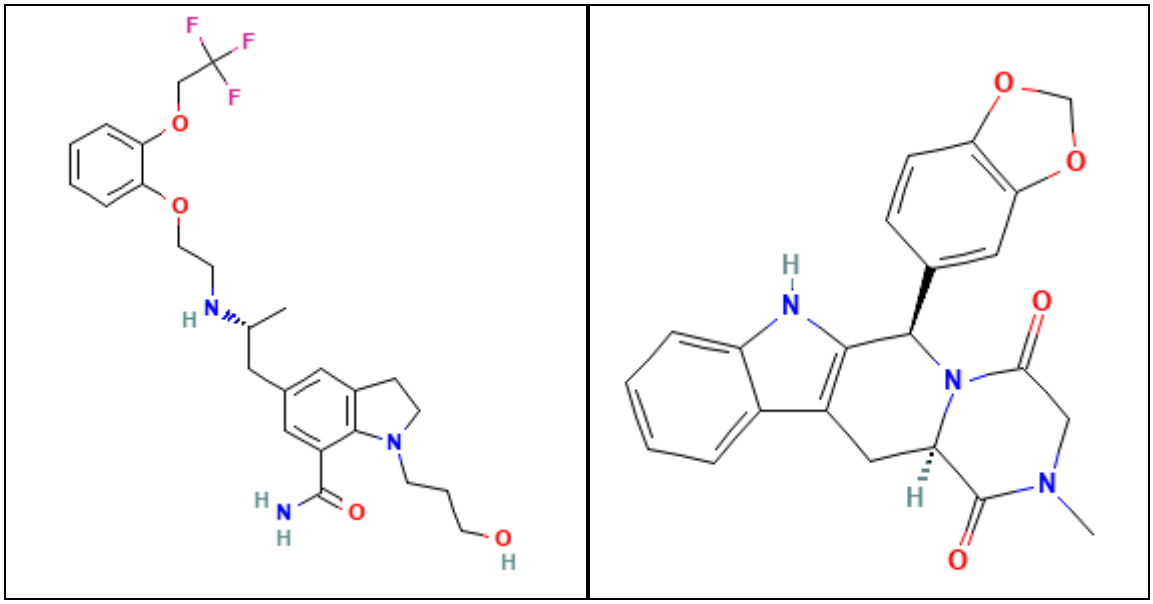

Figure 1 Structure of Silodosin and Tadalafil.

It appears your literature review shows that several analytical techniques, such as HPLC[6], HPTLC and spectroscopic techniques, have been employed to analyze drugs both individually and in combination. However, the specific combination of Silodosin and Tadalafil in capsule dosage form has not been estimated using stability in the literature so far. Therefore, an attempt will be made to develop stability method that is capable for simultaneous quantification of Silodosin and Tadalafil in capsule dosage form.

MATERIALS AND METHODS

Agilent technologies HPLC, Shimadzu UV-1900 UV-Visible spectrophotometer and Shimadzu IR Spirit X IR spectrophotometer instrument were used. Aglilent zorbax C_{18} (250 mm x 4.6 mm internal diameter x 5 µ particle size). Water, Acetonitrile, Dimethyl sulfoxide, Trifluoracetic acid, methanol of HPLC grade from Merck life SciPvt Ltd. / Finar Pvt. Ltd. / Aceto Pharma Pvt Ltd.

Selection of Mobile Phase

Various mobile phases were tried. Trial contains various mobile phases which consisted of acetonitrile, methanol, water, trifluoracetic acid and acetate buffer in different proportions with various pH and different volumes at flow rate 1 mL/min were tried. Chromatogram in optimized mobile phase is shown in Figure.

Solution Preparation

(A) Stock solution preparation of Silodosin

A 50 mg of Silodosin was weighed and transferred into a 100 mL volumetric flask. Volume was made up to the mark with DMSO.

(B) Working standard solution of Silodosin (160 ppm)

Further dilution takes 16 mL of stock solution and transferred into a 50 mL of volumetric flask. Volume made up to the mark with DMSO.

(C) Stock solution preparation of Tadalafil

A 50 mg of Tadalafil was weighed and transferred into a 100 mL volumetric flask. Volume was made up to the mark with DMSO.

(D) Working standard solution of Tadalafil (100 ppm)

Further dilution takes 10 mL of stock solution and transferred into a 50 mL of volumetric flask. Volume made up to the mark with DMSO.

(E) Mix Standard solution (Silodosin 160 ppm + Tadalafil 100 ppm): Further dilution take 10 mL of stock solution of Tadalafil and 16 mL of stock solution of Silodosin and transferred into a 50 mL of volumetric flask. Volume made up to the mark with DMSO.

(F) Buffer Preparation (Acetate Buffer pH 3.7): Dissolve 10 g of anhydrous sodium acetate in 300 ml of water, adjusted to pH 3.7 with glacial acetic acid and dilute with water to 1000 ml. Before use adjusted to pH 3.7, if necessary, with glacial acetic acid or anhydrous sodium acetate, as required.

(G) Buffer Preparation (Acetate Buffer pH 4.6): Dissolve 5.4 g of sodium acetate 50 ml of water, add 2.4 ml of glacial acetic acid and with water to 100 ml. Adjust the pH, if necessary.

(H) Buffer Preparation (Acetate Buffer pH 6.0): Dissolve 100 g of ammonium acetate in 300 ml of water, add 4.1 ml of glacial acetic acid, adjusted to pH, if necessary, using 10 M ammonia of 5 M acetic acid and dilute with water to 500 ml.

(I)Preparation of Solution A: Add 0.1 mL of trifluoro acetic acid to 1 L of water.

Forced Degradation Study

1. Acid Degradation

1 mL of filtered standard solution of Silodosin and Tadalafil was transferred into a 10 mL volumetric flask. Then 1.0 mL 0.1 N HCL solution was added, mixed thoroughly and kept at room temperature (25 °C) for 1 hrs. The solution was then neutralized with 1.0 mL 0.1 N NaOH solution and the volume was adjusted with the diluent to prepare the sample solution.

2. Base degradation

1 mL of filtered sample stock solution of Silodosin and Tadalafil was transferred into a 10 mL volumetric flask. Then 1.0 mL 0.1 N NaOH solution was added, mixed thoroughly and kept at room temperature (25 °C) for 1 hrs and 3 hrs. The solution was then neutralized with 1.0 mL 0.1 N HCl solution and the volume was adjusted with the diluent to prepare the sample solution.

3. Oxidative degradation

A 1 mL part of the stock solution of Silodosin and Tadalafil was transferred into a 10 mL volumetric flask. Then 1 mL 3% H2O2 solution was added, mixed thoroughly, and kept at room temperature (25 °C) for 1 hrs. Afterward, the volume was adjusted with the diluent to prepare the sample solution.

4. Photolytic Degradation

A 1 mL part of the stock solution of Silodosin and Tadalafil was transferred into a 10 mL volumetric flask. The solution was exposed to sunlight for 1 hrs and 8 hrs. Afterward, the volume was adjusted with the diluent to prepare the sample solution.

5. Thermal Degradation

Silodosin and Tadalafil were placed in petri dish and kept in hot air oven at 80 °C for 1 hrs. After the exposure, the samples were prepared for further analysis.

Method Validation[7]

Specificity

Specificity means accurately and precisely detecting the target analyte amid potential sample interferents like impurities, degradants and matrix elements. Proof must show spiked substances (impurities/excipients) do not interfere with results.

Linearity

It describes an analytical methods ability to yield results directly proportional to analyte concentration in samples (over a defined range).

Accuracy

This gauges closeness between measured values and accepted true/reference values.

Precision

Precision reflects consistency among repeated measurements of identical homogeneous samples under set conditions.

Repeatability

Precision under identical conditions over short time periods (also called intra-assay precision).

Intermediate precision

Captures lab-internal variations like different days, analysts, or instruments.

Detection Limit

LOD is the lowest analyte amount detectable (though not reliably quantifiable).

$$LOD = 3.3 \times (\sigma/S)$$

Quantitation Limit

LOQ marks the lowest analyte concentration reliably detectable and quantifiable with precision.

$$LOQ = 10 \times (\sigma / s)$$

Robustness

Robustness shows a method's capacity to remain reliable despite small, deliberate variations in parameters under routine use.

System suitability

System suitability tests form an integral part of method development. Criteria should align with the specific validation procedure type.

RESULT AND DISCUSSION

RP-HPLC optimized chromatographic condition

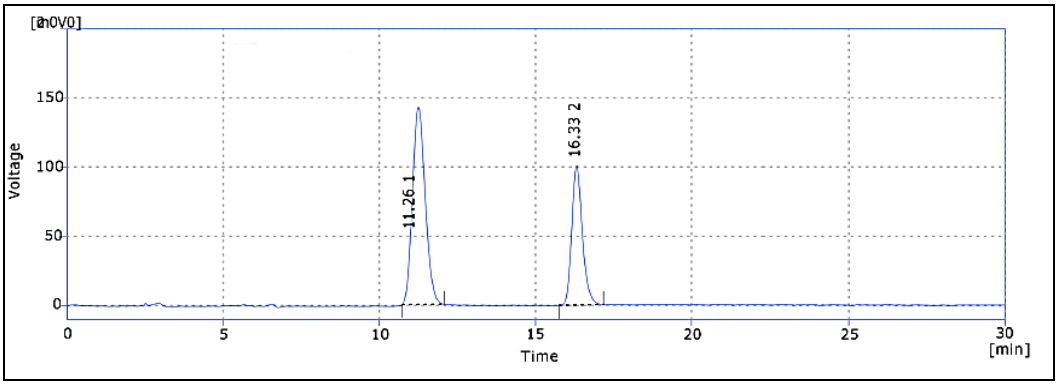

Figure 2: Chromatogram of Silodosin and Tadalafil in optimized condition.

Table 1: Optimized Chromatographic Conditions of Silodosin and Tadalafil.

Parameters	Chromatographic conditions
Elution mode	Isocratic
Stationary phase	Aglilent zorbax C_{18} (250 mm x 4.6 mm x 5 µ)
Mobile phase	Acetate buffer (ph 3.7): ACN: Trifluoracetic acid (55:43.5:1.5%v/v/v)
Concentration	Silodosin (160 ppm)+Tadalafil (100 ppm)
Flow rate	1 mL/min
Injection volume	20 µg/mL
Detection wavelength	284 nm
Column temperature	25 °C

Forced Degradation Study

1) Acid degradation

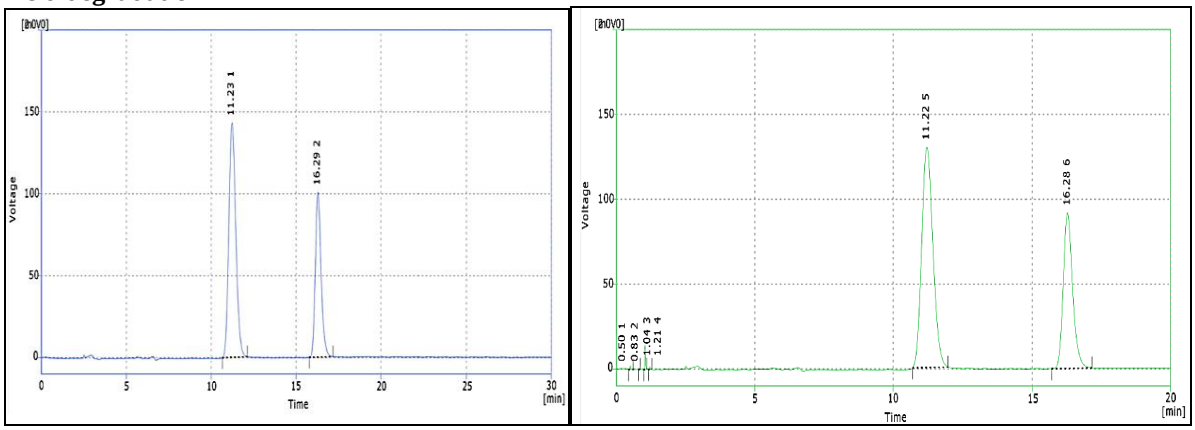

Figure 3 Chromatogram of Acid degradation Standard and Sample.

2) Base Degradation

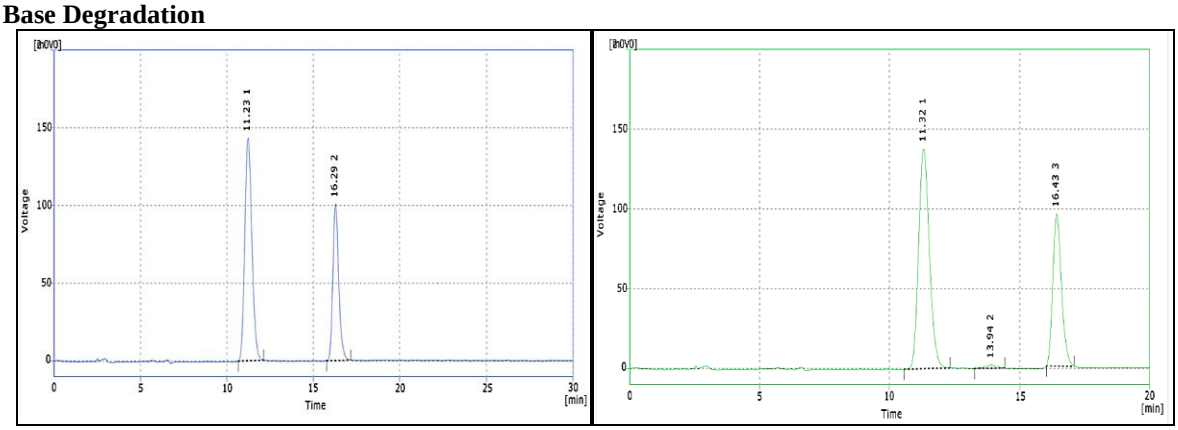

Figure 4: Chromatogram of Base degradation Standard and Sample.

3) Oxidative Degradation

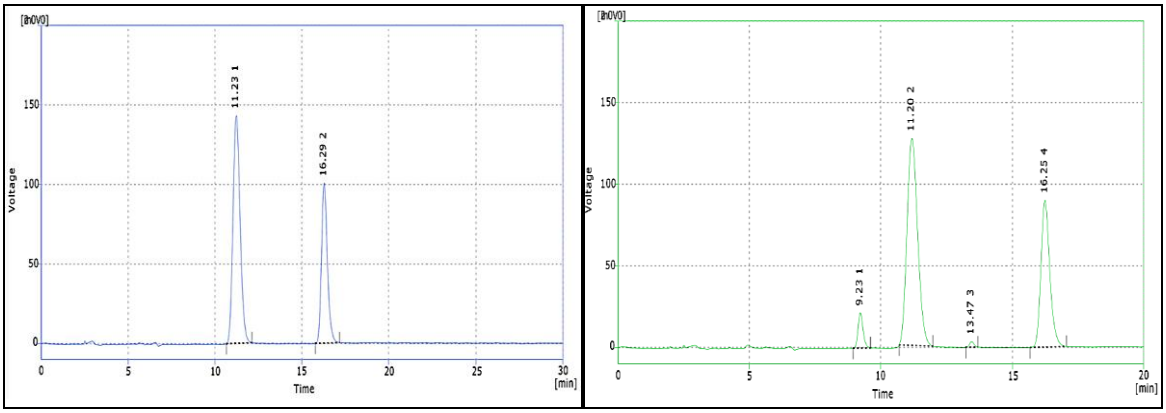

Figure 5: Chromatogram of Oxidative degradation Standard and Sample.

4) Photolytic Degradation

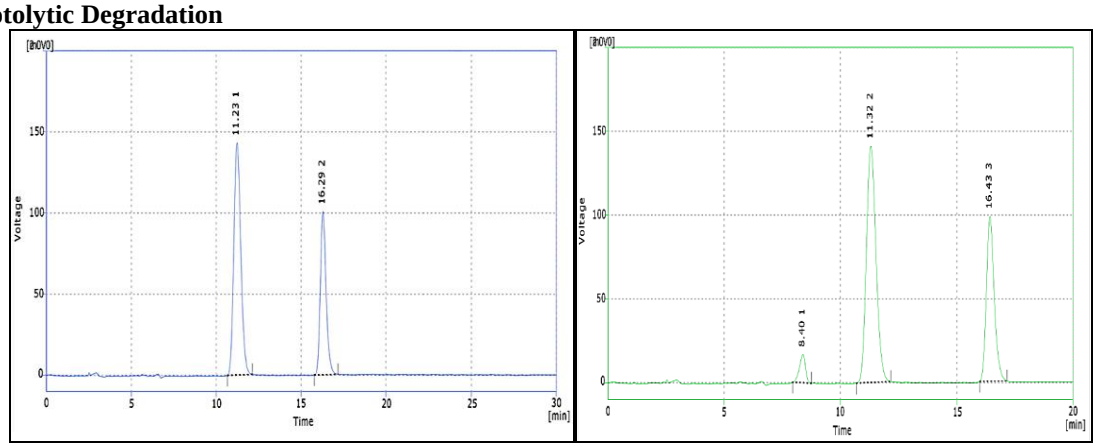

Figure 6: Chromatogram of Photolytic degradation Standard and Sample.

5) Thermal Degradation

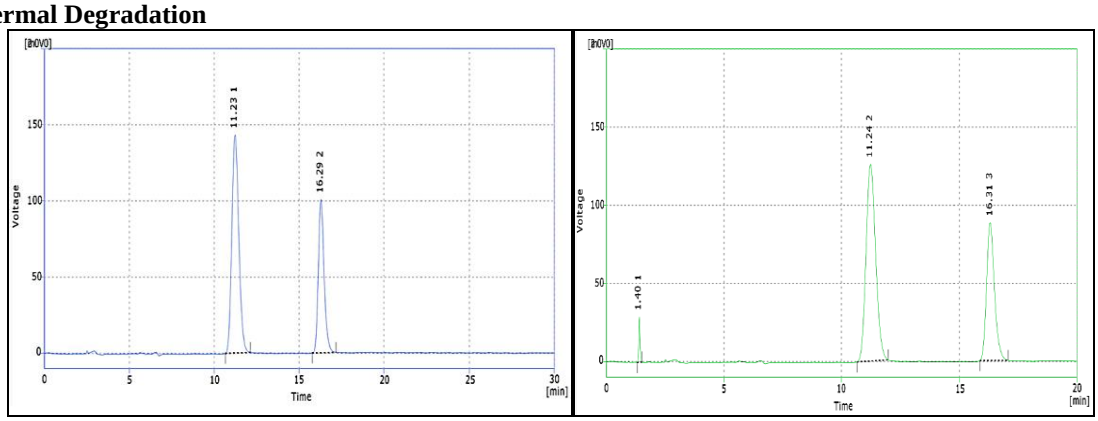

Figure 7: Chromatogram of Thermal degradation Standard and Sample.

Table 2: Forced Degradation Study.

Degradation study	Condition	Silodosin		Tadalafil	
		Area	% Degradation	Area	% Degradation
Acid degradation	0.1 N HCl for 1 hrs	3672.416	10.107	2161.86	8.773
Base Degradation	0.1 N NaOH for 1 hrs	3985.925	2.433	2212.858	6.621
	0.1 N NaOH for 3 hrs	3794.364	7.122	2195.261	7.364
Oxidative degradation	3 % H_20_2 for 1 hrs	3565.539	12.723	2110.936	10.922
Photolytic	Sunlight for 1	4055.669	0.726	2308.965	2.566

degradation	hrs				
	Sunlight for 8 hrs	3958.949	3.093	2289.896	3.370
Thermal degradation	80 °C for 1 hrs	3574.144	12.513	2058.709	13.126

Method validation

System suitability

System suitability was evaluated by injecting six replicate injections of the same concentration using the proposed method.

Table 3 System suitability for the replicated chromatographic condition for Silodosin and Tadalafil.

	Silodosin			Tadalafil		
Sr No.	**Area**	**Tailing factor**	**Theoretical plate**	**Area**	**Tailing factor**	**Theoretical plate**
1	2378.214	1.342	11182	4170.341	1.272	3440
2	2371.456	1.351	11167	4165.471	1.260	3430
3	2380.123	1.338	11195	4158.049	1.261	3487
4	2374.789	1.346	11174	4164.121	1.268	3453
5	2376.890	1.344	11188	4158.085	1.265	3459
6	2372.567	1.349	11170	4158.072	1.273	3430
Mean Area	2375.673			4162.356		
SD	3.456			5.133		
% RSD	0.145			0.123		

Specificity

The chromatogram of Silodosin and Tadalafil standards and Silodosin and Tadalafil sample show no involvement with the Chromatogram of Silodosin and Tadalafil blank, so the Developed method is specific.

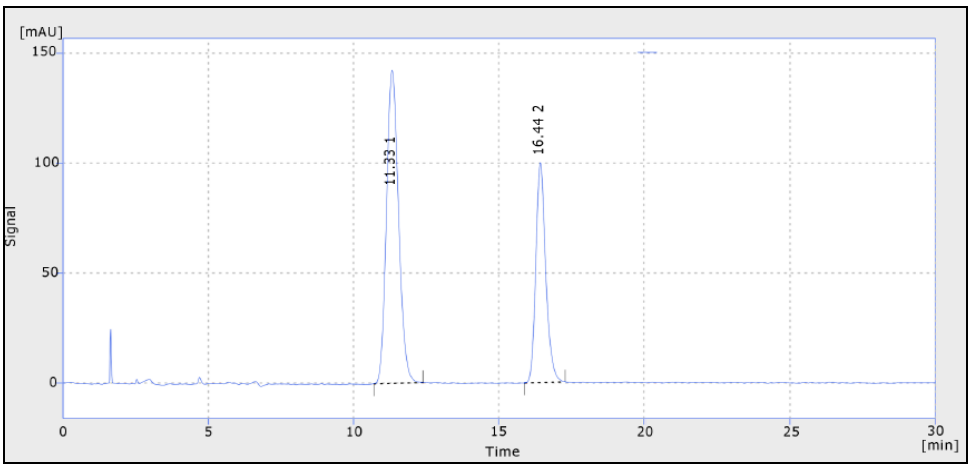

Figure 8: Chromatogram of sample.

Linearity

Table 4: Linearity data for Silodosin and Tadalafil.

Silodosin		Tadalafil	
Concentration (µg / mL)	**Mean area**	**Concentration (µg / mL)**	**Mean area**
80	1184.548	50	2054.406
120	1776.555	75	3071.302
160	2366.844	100	4114.643
200	2959.056	125	5116.626
240	3538.249	150	6117.127

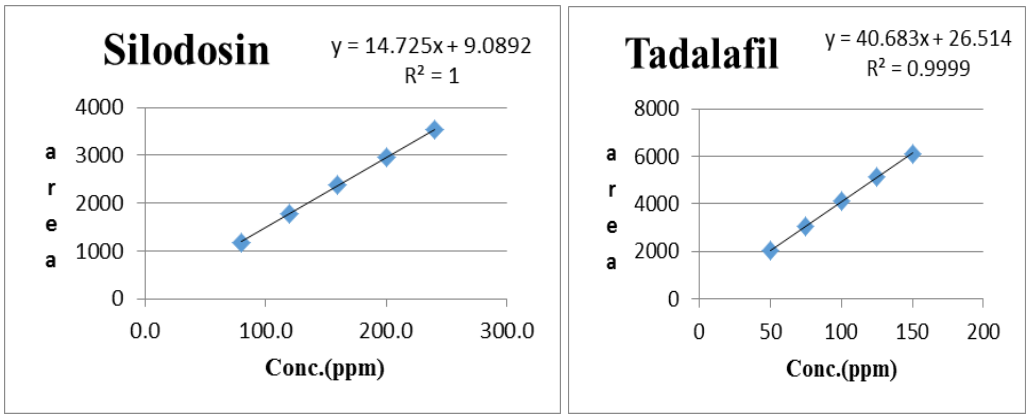

Figure 9: Calibration curve of Silodosin and Tadalafil.

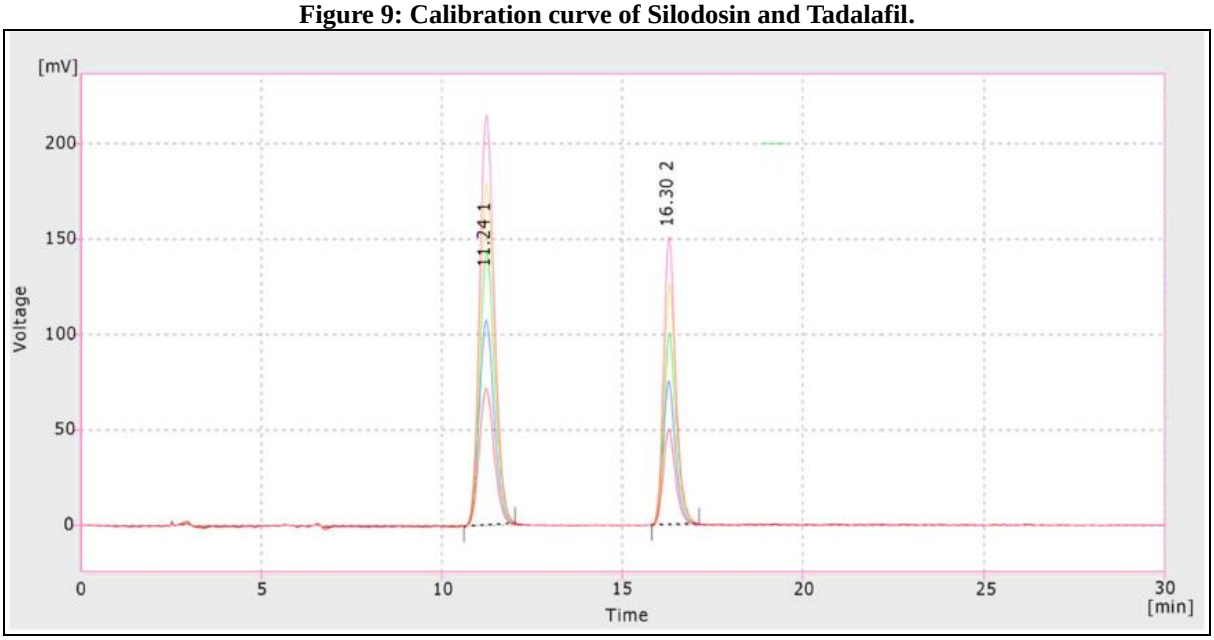

Figure 10: Overlay chromatogram of different concentration mixtures of Silodosin and Tadalafil.

Precision

Repeatability

The Repeatability studies were performed by measuring the response for a single concentration on six replicate injections within the same day.

Table 5: Repeatability data for Silodosin and Tadalafil.

Silodosin		Tadalafil	
Concentration	**Area**	**Concentration**	**Area**
160	2369.200	100	4118.321
160	2375.635	100	4120.684
160	2366.467	100	4115.295
160	2369.987	100	4110.063
160	2364.786	100	4117.420
160	2365.981	100	4123.213
Mean (n=6)	2368.676	Mean (n=6)	4117.499
SD	3.942	SD	4.553
% RSD	0.166	% RSD	0.111

Intraday and interday Precision studies were carried out by injecting six replicate sample solutions on the same day and on different days of analysis.

Table 6: Intraday Precision.

Siolodosin	Tadalafil

Conc. (µg / mL)	Area	% RSD	Conc. (µg / mL)	Area	% RSD
80	1182.807	0.232	50	2054.929	0.028
160	2364.549	0.056	100	4115.843	0.031
240	3538.368	0.073	150	6119.195	0.018

Table 7: Interday Precision.

Siolodosin			Tadalafil		
Conc. (µg / mL)	Area	% RSD	Conc. (µg / mL)	Area	% RSD
80	1185.139	0.058	50	2056.674	0.080
160	2363.289	0.133	100	4119.068	0.133
240	3537.560	0.064	150	6119.393	0.015

Accuracy

It was performed by the spiking the standard into placebo and calculating % recovery.

Table 8: Accuracy data of Silodosin and Tadalafil.

Sr. No.	Level	Silodosin			Tadalafil		
		Concentration spiked (µg / mL)	Concentration recovered (µg / mL)	% Recovery	Concentration spiked (µg / mL)	Concentration recovered (µg / mL)	% Recovery
1.	80%	128	127.93	99.95	80	80.15	100.19
2.		128	128.49	100.38	80	79.71	99.63
3.		128	127.54	99.64	80	80.24	100.31
1.	100%	160	160.62	100.39	100	100.38	100.38
2.		160	160.20	100.13	100	100.49	100.49
3.		160	161.53	100.96	100	100.12	100.12
1.	120%	192	191.85	99.92	120	120.07	100.05
2.		192	192.06	100.03	120	120.00	100.00
3.		192	192.62	100.32	120	120.59	100.49
Mean				100.19			100.19
± Standard Deviation				0.38			0.27
% Relative Standard Deviation				0.37			0.27

LOD and LOQ

Table 9: LOD and LOQ data.

	Silodosin	Tadalafil
Slope of Calibration curve	14.725	40.683
LOD (µg / mL)	0.984	1.181
LOQ (µg / mL)	2.982	3.580

Robustness

The method was evaluated by intentional variation of different parameters, and the results remained within the specified acceptance limits. The percentage relative standard deviation (%RSD) was found to be less than 2%, indicating that the method is robust and resistant to small changes in experimental conditions.

Table 10: Robustness data for Silodosin and Tadalafil.

Drug	Area at Flow rate (-2 mL/min)	Area at Flow rate (+2 mL/min)	Area at Mobile Phase (-2 mL/min)	Area at Mobile Phase (+2 mL/min)	Area at pH (-2)	Area at pH (+2)
Silodosin	2391.582	2498.349	2246.112	2567.219	2424.246	2436.521
	2382.092	2486.950	2275.804	2582.649	2419.873	2448.291
	2397.398	2504.678	2264.978	2549.422	2422.689	2455.399
Mean	2390.357	2496.659	2262.298	2566.430	2422.269	2466.737
SD	7.726	8.984	15.026	16.628	2.217	9.534
% RSD	0.323	0.360	0.664	0.648	0.092	0.390
Tadalafil	4194.542	4215.253	4026.465	4365.954	4210.389	4299.321
	4189.231	4264.562	4065.901	4349.342	4219.584	4267.029
	4187.697	4235.157	4078.420	4337.985	4215.607	4284.444
Mean	4190.490	4238.324	4056.929	4351.094	4215.193	4283.598
SD	3.592	24.807	27.115	14.067	4.611	16.163
% RSD	0.086	0.585	0.688	0.323	0.109	0.377

Assay

The applicability of the proposed method was evaluated by analysing a synthetic mixture. The results, expressed as percentage assay, are presented in the following table.

Table 11: Assay of Silodosin and Tadalafil.

Sr. No.	Silodosin		Tadalafil	
	Area of Sample	**% Assay**	**Area of Sample**	**% Assay**
1.	2373.063	100.294	4121.643	100.613
2.	2368.172	100.087	4105.251	100.213
3.	2351.828	99.396	4079.731	99.590
	Mean	99.926	**Mean**	100.138
	Standard Deviation	0.470	**Standard Deviation**	0.516
	% RSD	0.470	**% RSD**	0.515

CONCLUSION

A stability indicating RP-HPLC method was successfully developed and validated for the simultaneous estimation of Silodosin and Tadalafil in Capsule dosage form. The method is simple and economical, employing an easily available mobile phase. It provides high sensitivity, as indicated by low limits of detection and quantitation for both analytes, along with a short analysis time, making it well suited for routine quality control. The method uses isocratic elution, which improves simplicity and ease of operation without compromising performance. It has been validated in accordance with ICH guidelines and demonstrates excellent precision, accuracy, specificity and robustness. Overall, the developed method is accurate, specific, robust, fast and cost-effective and therefore highly suitable for regular quality assessment.

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