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Development and Validation of Stability Indicating HPLC Method for Estimation of Dapagliflozin in Marketed Formulation

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ABSTRACT

To develop precise, accurate, reproducible and validate stability indicating HPLC method for determination of Dapagliflozin in API and pharmaceutical formulation as per ICH Q2 R1 Guidelines. The adequate separation was carried using mixture of methanol: water (80: 20% v/v) as a mobile phase with the flow rate of 0.8 ml/min and the effluent was monitored at 225nm using single wavelength UV detector. A stability-indicating HPLC method has been developed for analysis of the drug in the presence of the degradation products and is validated with different parameters such as linearity, Precision, Accuracy, Robustness and Ruggedness. It involved various time interval study in which methanol and distilled water were used as solvents. Beer's law was obeyed in the concentration range of 10-50 µg/ml. A retention time of Dapagliflozin API and tablet were 6.744 min and 6.69 min respectively. A recovery of Dapagliflozin in tablet formulation was observed in the range of 99.76-100.01%. Degradation of Dapagliflozin was found to occur in acid, alkaline, hydrogen peroxide and thermal conditions whereas it was found to be thermally stable. The proposed method was found to be specific, accurate, precise and robust can be used for estimation of dapagliflozin in API and pharmaceutical formulation.

INTRODUCTION

Stability testing and stress degradation studies play a very crucial role in drug development. Stability is fundamental to all product characteristics, and the term "Stability indicating assay" has been used to describe a procedure which affords specific determination of drug substance in the presence of its degradation products. The prime goal of studying the stability of a drug is to determine the shelf life of the drug. The various conditions specified for stress degradation studies include acidic, alkaline, oxidation, photolytic and thermal.

Dapagliflozin belongs to a new class of oral antidiabetic drugs, called Sodium Glucose Co-Transporter 2 (SLGT2) inhibitors. These sodium glucose co-transporters are responsible for glucose reabsorption in the kidney. Hence inhibiting the SLGT2 have been proposed as a new strategy in the treatment of diabetes.[1,2,3] suppressing the SLGT2, dapagliflozin plasma glucose concentration intern by elevating the renal glucose excretion by the kidney.

It is chemically known as (1s)-1,5- anhydro-1-C-[4-chloro-3-[(4-ethoxy-phenyl)methyl]phenyl]-D-glucitol(fig.1).It has a molecular formula $C_{24}H_{33}CLO_8$ with molecular weight 408.98. Dapagliflozin is a white crystalline powder which is soluble in water, ethanol, methanol and dimethylformamide.(1-7).

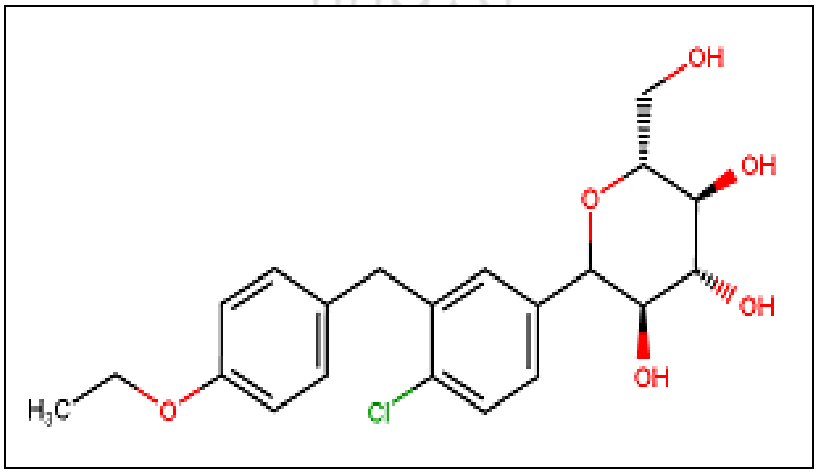

Figure No: 1 Chemical structure of Dapagliflozin

AIM AND OBJECTIVES

A thorough literature survey has revealed that there are few analytical methods reported for quantitative estimation of Dapagliflozin and its stability study. In the present work a successful attempt has been made to develop accurate, precise, economical and rapid analytical method for estimation and its stability study is done by HPLC.

LITERATURE REVIEW:

1) Jitendra Kumar et al**:** have reported that forced degradation studies show the chemical behavior of the molecule which in turn helps in the development of formulation and package. A forced degradation study is an essential step in the design of a regulatory compliant stability program for both drug substance and product, and formalized as a regulatory requirement in ICH guideline Q1A in 1993. Forced degradation is a degradation of new drug substance and drug product at condition more severe than accelerated conditions. Forced degradation studies providing a strategy for conducting studies on degradation mechanisms.(8)

2) Shubhangi et al: have reported the stability of drug product or a drug substance is a critical parameter which may affect purity, potency and safety. Forced degradation studies show the chemical methods for quantification of dapagliflozin in bulk and its tablets. The developed methods were validated for accuracy, precision, ruggedness and sensitivity as per ICH guidelines. Detect possible degradants and impurities of drug substance (API) and drug products, normally using High Performance Liquid Chromatography. (9)

3) Manasa et al**:** have reported two analytical methods developed for the estimation of Dapagliflozin in API. RP-HPLC method and UV spectroscopic method. In RP-HPLC method the drug show linearity in the range of 25-150µg/ml with a correlation coefficient (r2) of 0.999,in UV spectroscopic method the linearity range was found to be 1-5µg/ml with a correlation coefficient of (r2) 0.999.(10)

4) Manasa Sanagapati et al: have reported the development and subsequent validation of a stability indicating reverse phase HPLC (RP-HPLC) method for the analysis of Dapagliflozin in its API. The proposed method utilizes BDS column (maintained at ambient temperature), gradient run (using mixture of acetonitrile and orthophosphoric acid as mobile phase), effluent flow rate (1ml/min) and detection at 245nm using PDA detector.The developed

method was successfully validated as per ICH guidelines. The stability of the drug was determined by studying the degradation of the drug under acidic, alkaline, peroxide, neutral, heat and UV conditions.(11)

5) Trivikram Rawat et al: have reported that forced degradation is the process to facilitate the development of analytical methodology, to gain better understanding of API and drug product stability, and to provide information about degradation pathway and degradation products. This publication provides information about regulatory needs and scientific guidance to perform forced degradation.(12)

6) Mitali Verma et al: have reported precise, accurate and reproducible stability assay method by RP-HPLC method for estimation of dapagliflozin in API and pharmaceutical dosage form. The adequate separation was carried using Agilent C18 [4.6 ml (millimeter) 150,5µm (micrometer), mixture of acetonitrile : dipotassium hydrogen phosphate with pH-6.5 adjusted with OPA [40:60% v/v] as a mobile phase with the flow rate of 1 ml/ min and the effluent was monitored at 222nm using photodiode array detector. The retention time of dapagliflozin API and dapagliflozin tablet were 3.160 min and 3.067 min respectively.(13)

7) Khyati J. Patel et al: have reported to Stability indicating RP- HPLC method development and validation for estimation of Dapagliflozin and Metformin HCL. A simple, specific, accurate, precise and reproducible and robust method has been developed and validated for the simultaneous estimation of dapagliflozin and metformin HCl in RP- HPLC method. The separation was carried out using initial ODS C18 column (250mm × 4.6 mm, 5µ) as stationary phase and UV detector set at 227 nm, in conjunction with a mobile phase of 0.05 M potassium dihydrogenortho phosphate buffer (PH– 3.5, adjusted with 0.1 % orthophosphoric acid) and acetonitrile in the ratio of 50:50% v/v at flow rate 1.0ml/min. The % recovery was found to be 100.40%- 101.27% respectively. So, the method can be used for routine analysis.(14)

8) Phani RSCH et al: have developed a simple, precise and stability indicating reversed phase High Performance Liquid Chromatography (RP-HPLC) method for simultaneous quantification of dapagliflozin and saxagliptin in combined dosage form. The developed method has been validated with respect to precision, linearity, accuracy, robustness, ruggedness, sensitivity, solution stability. The method has been developed with ammonium dihydrogen phosphate buffer (pH 6.8) and methanol in a ratio of 65:35 v/v as mobile phase at

a flow rate of 1.5 ml/min over intersil ODS C18 column (250 mm × 4.6 mm × 5µ). The UV wavelength was fixed at 280 nm. The method show good linearity with correlation coefficient values of 0.9992 and 0.999 for DGFZ and SGPT. The % recoveries of two drugs found within the limits of (98.00-102.0%). According to international conference on Harmonization (ICH) guidelines forced degradation study was validated. (15)

MATERIALS AND METHODS

Materials:

Dapagliflozin was procured as gift samples from Indico Remedies, Mumbai. The HPLC system employed was Analytical technologies Pvt. Ltd. HPLC instrument equipped with series 3000M UV/ Visible detector, pump model no. P-3000M, 40MPa maximum pumping capacity, chromatography interface 3000 series link. (Nashik, India), coming volumetric flasks and pipettes of borosilicate glass were used in the study. Methanol AR Grade was procured from S.D. Fine Chemicals Ltd., Mumbai, India. Whatman filter paper No. 41 (England) was also used in the study. The solution of 0.1 N NaOH was prepared in double distilled water as per IP 1996 procedure.0.1 N HCl was prepared following the procedure outlined in BP 1999 and USP 24.

Preparation of standard stock solution and selection of detecting wavelength:

1] Standard stock solution:

An accurately weighed quantity of about 10 mg of Dapagliflozin was taken in 10.0 ml volumetric flask, dissolved in HPLC grade methanol and volume was made up to mark with same solvent (conc. 1 mg/ml). The aliquot portion of the standard stock solution was diluted appropriately with the same solvent to obtain the concentration of 10µg/ml. This solution was scanned in 1 cm cell using double beam UV- Visible Spectrophotometer over the range of 400-200 nm and the UV absorbance spectrum was recorded. From the spectrum, the detecting wavelength selected for estimation of the drug was 225.0 nm as shown in **Figure No: 2**

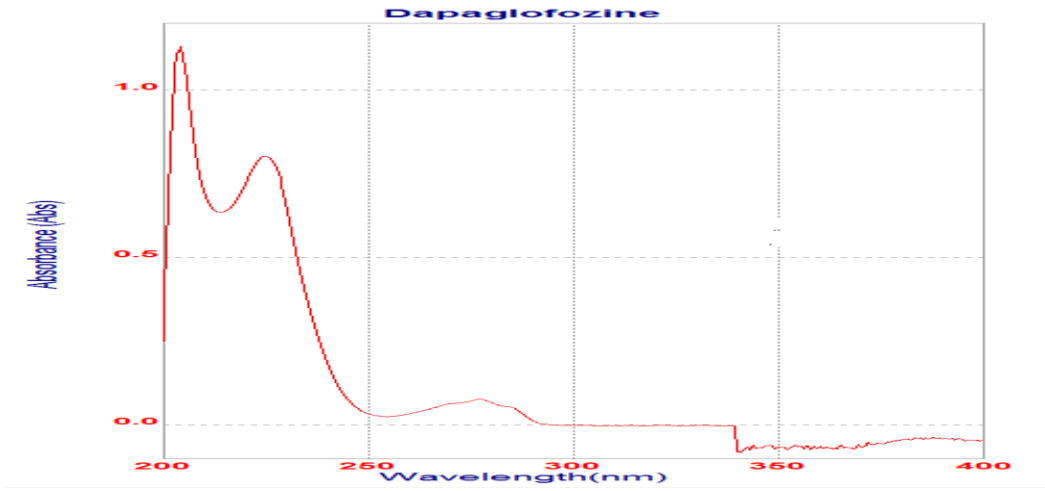

Figure No: 2 UV absorption spectrum of Dapagliflozin

2] Selection of mobile phase

The pure drug of Dapagliflozin was injected into the HPLC system and run in different solvent systems. Each mobile phase was allowed to equilibrate with stationary phase until steady baseline was obtained. Different mobile phases like methanol and water, acetonitrile, and water in various proportions were tried. Different individual solvent, as well as combinations of solvents, were tried to get a stable peak. Each mobile phase was filtered through 0.45µm nylon membrane filter and sonicated on the ultrasonic bath. After several trials methanol: water (80:20 v/v) was found to be the most satisfactory since it gave sharp, peak with symmetry within limits and significant reproducible retention time.

Table No: 1 Selection of mobile phase

Sr. No.	Mobile phase strength (v/v)	Rt of Dapagliflozin [min]	Conclusion
1	Methanol 100%	2.01	Peak not satisfactory
2	Water 100%	7.05	Peak not satisfactory
3	Methanol:water(50:50)	4.02	Peak not satisfactory
4	Methanol: water (80:20)	6.74	Peak satisfactory

3] Selection of chromatographic parameters

The method was developed using C_{18} Inertsil (250× 4.6 mm) column. The mobile phase used was methanol: water (80:20v/v). Flow rate adjusted was 0.8 mL/min. Detection was carried out at 225.0 nm. The mobile phase and samples were degassed by ultrasonic vibrations for 20

min. and filtered through 0.45µm nylon membrane filter. All determinations were performed at constant temperature.

4] Preparation of mobile phase and construction of calibration curves

Mobile phase was prepared by mixing 800 mL of methanol with 200mL of water. The mobile phase was sonicated for 15min on ultrasonic bath and then it was filtered through 0.45µm nylon membrane filter.

The aliquot portion of the standard stock solution was diluted appropriately with same to obtain concentration of 100 µg/mL. The mobile phase was allowed to equilibrate with stationary phase until steady baseline was obtained. The appropriate aliquot of Dapagliflozin stock solution was transferred in series of 10.0 mL volumetric flask and volume was made up to the mark with mobile phase to obtain the various concentration of 10- 50 µg/ mL. These solutions were injected using a 20µL fixed loop system separately in triplicate and chromatographed under conditions described above and peak areas were recorded. The graph was plotted as concentration of drug Vs peak area and depicted in **Figure No: 3**

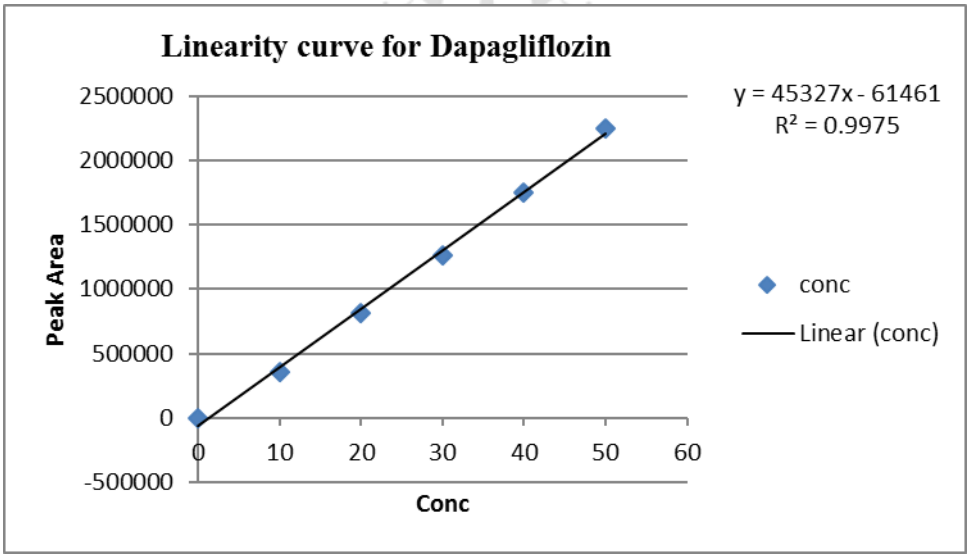

Figure No: 3 Calibration curve for Dapagliflozin

Table No: 2 Final chromatographic conditions.

Chromatographic mode	Chromatographic conditions
Standard solution	20 µg/mL of Dapagliflozin in mobile
Stationary phase	C_{18} (5 µm, 250 mm X 4.6 mm i.d.)
Mobile phase	Methanol: Water(80:20)
Detection wavelength	225 nm for Dapagliflozin
Flow rate	0.8 mL/min
Column temperature	Ambient
Detector& pump No.	UV 3000 M & P3000 M
Software	HPLC Workstation
Sample Size	20µL

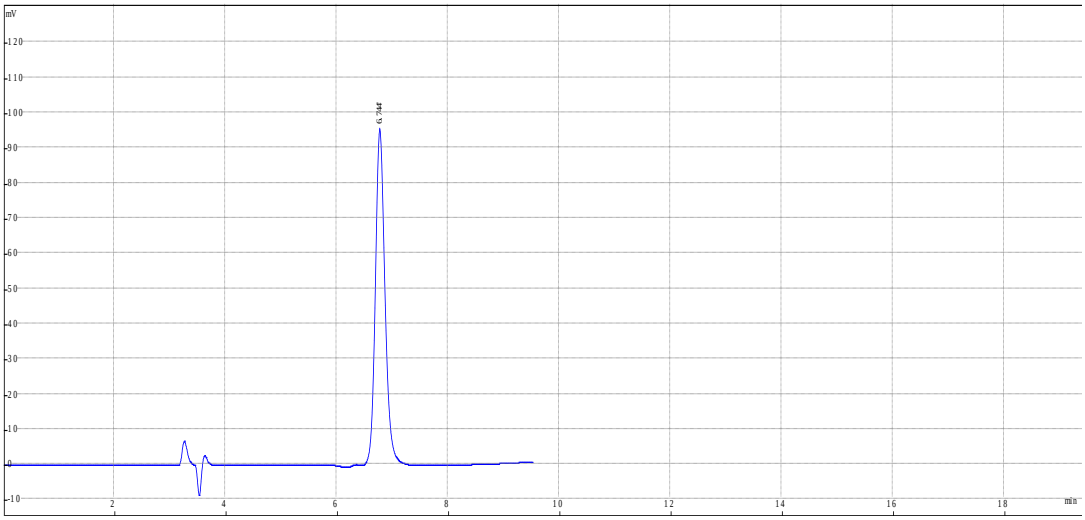

Figure No. 4 Typical chromatogram of Dapagliflozin (RT 6.744 respectively)

5] System suitability Parameters

System suitability test is a pharmacopoeial requirement and is used to verify, whether the resolution and reproducibility of the chromatographic system is adequate for analysis to be done. The tests were performed by collecting data from five replicate injections of standard drug solution.

Previously filtered mobile phase was allowed to equilibrate with stationary phase until baseline was achieved. 20µL standard drug solution was injected separately and their system suitability parameters were recorded.

Table No: 3 Observations of system suitability parameters

Sr. No.	Area Reproducibility	Retention time	Tailing Factor	Theoretical Plate	% area
1	358399	6.703	1.15	5017	100.00
2	812187	6.678	1.19	6592	100.00
3	1261832	6.698	1.19	6195	100.00
4	1749738	6.671	1.18	6495	100.00
Mean		**6.6875**	**1.1775**	**6074.75**	
Limit	--------	-----	**NMT 2**	**NLT 2000**	

6] Application of the proposed method to standard laboratory mixture

The laboratory mixture was prepared in the ratio of 80:20 v/v for Dapagliflozin respectively**.**

Details of marketed formulation

Trade Name: Forxiga

MFG: AstraZeneca

Content: 10mg/tablet

Avg. wt.: 258.83mg

1] Standard solution

Accurately weighed quantity of 10mg Dapagliflozin was transferred to 10mL volumetric flask volume was adjusted up to the mark with HPLC grade methanol mobile phase. The solution was then filtered through a 0.45µm-membrane filter. Accurately measured 1.0 mL portion of filtrate was further diluted to 10.0mL with mobile phase (10µg/mL).

2] Sample solution

Twenty tablets were accurately weighed and average weight was calculated. Tablets

Were finely powdered and mixed thoroughly. An accurately weighed powder equivalent to about 10 mg of Dapagliflozin was transferred to 10.0 mL volumetric flask. The content was shaken for 10-15 minutes with HPLC grade methanol mobile phase and volume was adjusted up to mark with same. The solution was then filtered through a 0.45µm membrane filter.

Accurately measured 1.0 mL portion of the filtrate was further diluted to 10.0 mL with mobile phase. Equal volumes (20µL) of std and sample solutions were injected separately after equilibrium of stationary phase. The chromatogram was recorded and the response that is peak area of major peak was measured. The content of Dapagliflozin was calculated by comparing a sample peak with that of the standard.

Typical chromatogram of standard and sample are shown in **Figure No: 5**

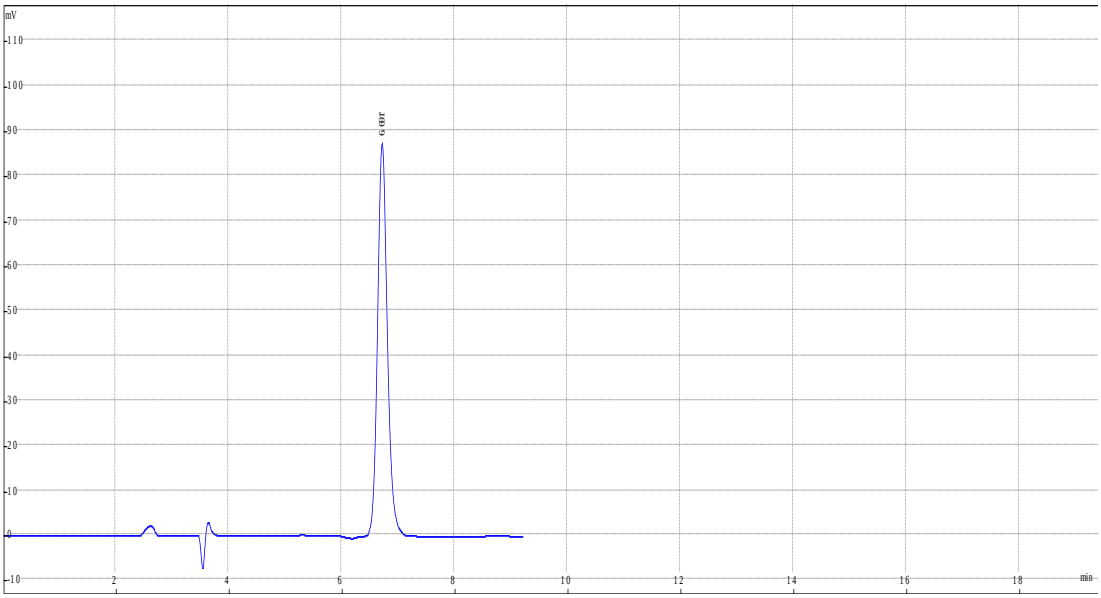

Figure No: 5 Typical chromatogram of Dapagliflozin sample (marketed formulation) (RT= 6.69)

Amount of drug in tablet was calculated using formula

$$\textbf{\% Label claim} = \frac{At}{As} \times \frac{Ds}{Dt} \times \frac{Ws}{Wt} \times \frac{A}{LC} \times 100$$

Where,

A = Average weight

At = Area for sample solution

As = Area for standard solution

Ds = Dilution factor for standard solution

Dt = Dilution factor for sample solution

Ws = Weight for standard solution

Wt = Weight for sample solution

LC = Label claim

Five replicate estimations were done in similar way. Observations and results in marketed formulation are shown in **Table No: 4**

Table No: 4 Observations and results of % label claim of Dapagliflozin

Tablet	**Weight of tab powder [µg/mL]**	**Peak area**	**% Label claim**
Std. Sample	1.0	358399	100.68
	25.88	368297	100.08
	26.08	368496	101.47
	26.21	358399	100.55
Test sample	26.40	379994	98.22
	25.88	398499	100.37
		Mean	100.22
		±S.D	1.0885
		%R.S.D	1.0861

7] Validation

Validation of proposed method was carried out for the following parameters as per USP guidelines.

1] Accuracy

Accuracy of the proposed method was ascertained on the basis of recovery studies performed by standard addition method.

Recovery studies

It was carried by standard addition method.

Sample solution

Accurately weighed quantities of preanalysed tablet powder equivalent to 10 mg Dapagliflozin was taken in 10.o ml volumetric flask and then known amount of Dapagliflozin was added at different concentration levels so as to produce solutions containing 80%, 100% and 120% of the label claim. The contents of the flasks were shaken with HPLC grade methanol volumes were adjusted up to the mark. The solutions were filtered through a 0.45µm- membrane filter. Accurately measured 1.0 mL portion of each filtrate was diluted to 10.0mL with mobile phase. The amount of drug contributed by the tablet powder was deducted from the total amount of estimated and the resultant quantity was assumed to be recovered from the added pure drug. The content of drug was calculated using the same formula as in

Observations, results and statistical data for recovery studies are shown in **Table No. 5**

Table No: 5 observations, results and statistical data for recovery studies

Sr. no.	Wt. of tablet powder (mg)	Peak area of std	Amount of pure drug added µg/ml	Peak area of sample	% recovery
1	256.86	4717390.66	8	4716960	99.99
2	259.16		10	4717998.33	100.01
3	256.9		12	4706341.33	99.76
				Mean	99.92
				± S.D	0.1389
				%R.S.D	0.1390

2] Precision

Precision of an analytical method is expressed as S.D. or R.S.D of series of measurements. It was ascertained by replicate estimation of the drugs by proposed method for marketed fore shown in mulation.

3] Linearity and Range:

Accurately weighed quantities of tablet powder equivalent to about 60, 70, 80, 90 and 100% of label claim was taken and dissolved in methanol, diluted appropriately to obtain a concentration in the range of 60% - 100% of test concentration. The absorbances of the resulting solutions were recorded at 225 nm. **Figure No: 6**

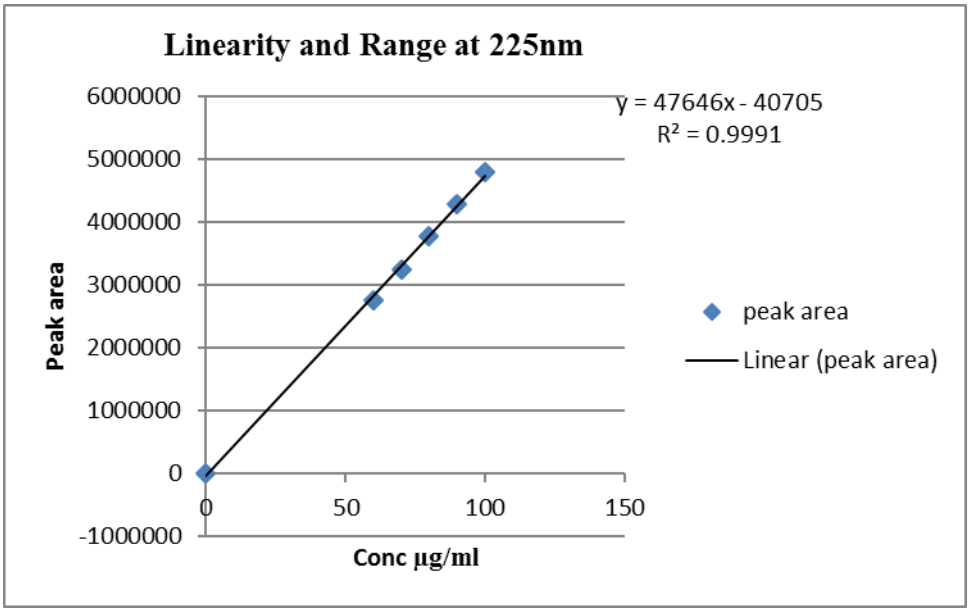

Fig No: 6 Linearity and range of Dapagliflozin at 225 nm

4] Ruggedness

The ruggedness of the method was studied under three different parameters. Samples were prepared as per marketed formulation analysis.

a] Inter –day Result

The samples were analyzed by proposed method on three different days (1^{st}, 2^{nd}, 3^{rd} day). The percent labeled claim was calculated and results of estimation are shown in **Table No: 6**

Table No: 6 Results and statistical data for Inter-day study

Day	Wt. of tab powder taken (mg)	Wt.ofstd (mg)	Std peak area	Sample peak area	% Label claim
1	258	10	358399	358297	100.29
2	257	10	368297	369190	100.95
3	257.4	10	358399	354061	99.33
			Mean		**100.19**
			±SD		**0.8146**
			%R.S.D		**0.8130**

b.]Intra -day Results

The samples were analyzed on different times on same day by proposed method. The percent labeled claim was calculated and results of estimation are shown in **Table No.7**

Table No: 7 Results and statistical data for interday study

Time (hr)	Wt. of tab powder taken (mg)	Wt. of std (mg)	Std peak area	Sample peak area	% Label claim
1	256.5	10	358399	358399	100.90
2	258	10	379297	376282	99.52
3	258.6	10	398690	398499	100.04
			Mean		**100.15**
			±S.D		**0.6969**
			% R.S.D		**0.6958**

c] Different analysts

The samples were analyzed by three different analysts as per the proposed method. The percent labeled claim was calculated and results of estimation are shown in**Table No: 8**

Table No: 8 Results and statistical data for different analyst study

Analysts	Wt. of tab powder taken (mg)	Wt. of std (mg)	Sample peak area	Sample peak area	% Label claim
Analyst-1	258.3	10	358399	358199	100.14
Analyst -2	258.8	10	367890	365070	99.24
Analyst -3	258	10	398499	397499	100.06
			Mean		**99.81**
			±S.D		**0.4981**
			% R.S.D		**0.4990**

8] Preparationof stress degradation sample (16-19)

Preparation of std solution

The solution was prepared as described under preparation of std solution (1000µg/ml)

Preparation of sample solution

An accurately weighed amount of tablet powder equivalent to 10.0mg of Dapagliflozin was separately dissolved in 10.0 ml of HPLC grade mobile phase methanol solution of 0.1 N HCl, 0.1 N NaoH, and 5% H_2O_2. From above solution 1 mL was pipette out and transfer into 10mL volumetric flask and 1 mL of 0.1 N HCl was added and refluxed at 60^0c for about 30 min and the same procedure repeat for 0.1 N NaOH. From above solution 1 mL was pipette out and transferred into 10mL volumetric flask and 1mL of 5% H_2O_2 was added and refluxed at 60^0C for 12 hr. Then 1.0 ml of above solutions were taken, neutralized and diluted up to 10.0 ml with water. Photolytic degradation was carried out by exposing the drug solution to sunlight for 30 min. Thermal degradation was carried out by exposing the sample in an oven at 60^0C for 30 minutes. 0.1N NaOH. After time period cool the contents to ambient temperature. Make up the volume with water.

Stress Degradation Studies

Forced degradation studies were performed to evaluate the stability indicating properties and specificity of the method. Dapagliflozin degraded in hydrolytic conditions as its percentage

degradation was found. The degradation parameter was optimized for hydrolytic stress condition were followed for producing stress degradant sample. **Table no. 9**

Table No: 9 Optimized stress condition.

Sr.No	Stress Condition	Optimum stress condition	Time
1	Acidic Hydrolysis	0.1N HCl refluxed at 60^{0}C	30 min MIN
2	Basic Hydrolysis	0.1N NaOH refluxed at 60^{0}C	30 min
3	Hydrogen peroxide	5% hydrogen peroxide refluxed at 60^{0}C	12 hr
4	Thermal	Drug sample placed in Oven at 60^{0}C	30 min
5	Photolytic	Drug sample exposed by Sunlight	30 min minimum

1] Acidic

To 1 mL of stock solution, 1 mL of 0.1 N HCl was added and refluxed at 60^{0}C for about 30 minutes. Then the resultant solution was sufficiently diluted to get 100µg/mL solution. From this, 20µl was injected into the system. From the peak area found in the chromatogram, the % degradation was calculated.

Dapagliflozin is found to be degraded in acidic condition. As shown in **Figure No.7**

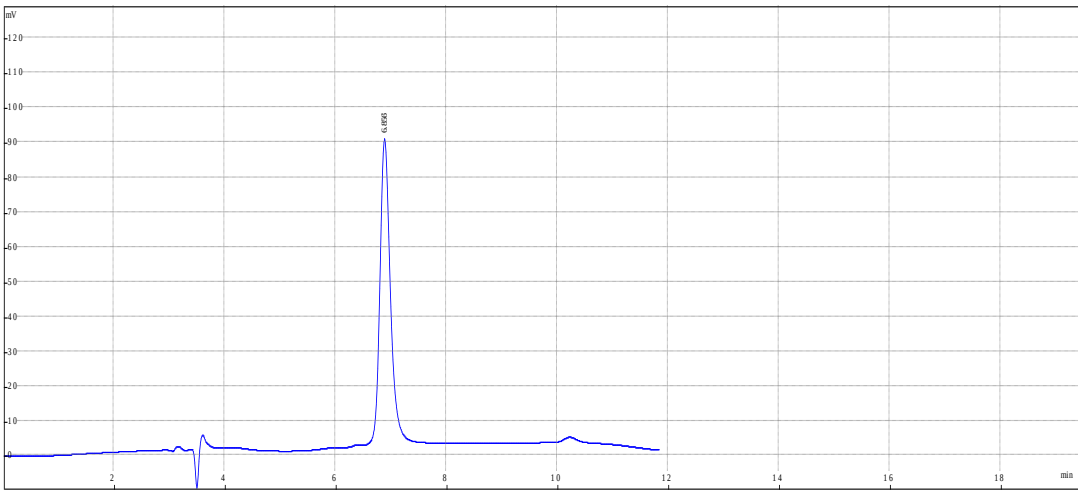

Figure No: 7 Chromatogram for Acidic Degradation Study

Time	Area	Resolution	T. platinum	Asymmetry
6.856	1025260	0.00	5527	1.27

2] Alkaline degradation

To 1 mL of stock solution, 1 mL of 0.1 N NaOH was added and refluxed at 60^0c for about 30 minutes. Then the resultant solution was sufficiently diluted to get 100µg/mL solution. From this, 20µl was injected into the system. From the peak area found in the chromatogram, the % of recovery was calculated. Dapagliflozin is found to be degraded in alkaline condition. As shown in **figure No. 8**

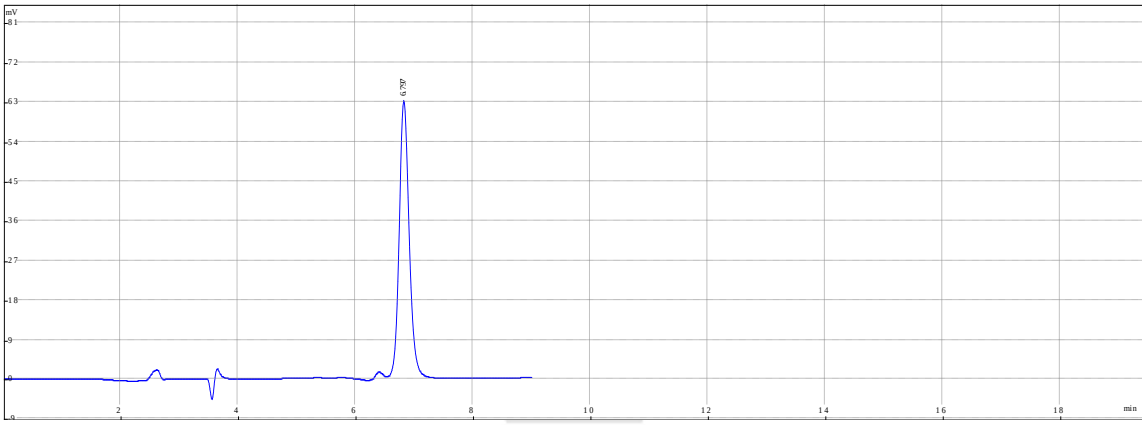

Figure No: 8 Chromatogram for alkaline degradation

Time	Area	Resolution	T. platinum	Asymmetry
6.797	868132	0.00	7398	1.14

3] Oxidative degradation

To 1 mL of stock solution, 1 mL of 5% H_2O_2 was added and refluxed at 60^0c for about 12 hrs. Then the resultant solution was sufficiently diluted to get 100µg/mL solution. From this, 20µl was injected into the system. From the peak area found in the chromatogram, the % of recovery was calculated.

Dapagliflozin is found to be degraded in Oxidative condition. As shown in **figure No. 9**

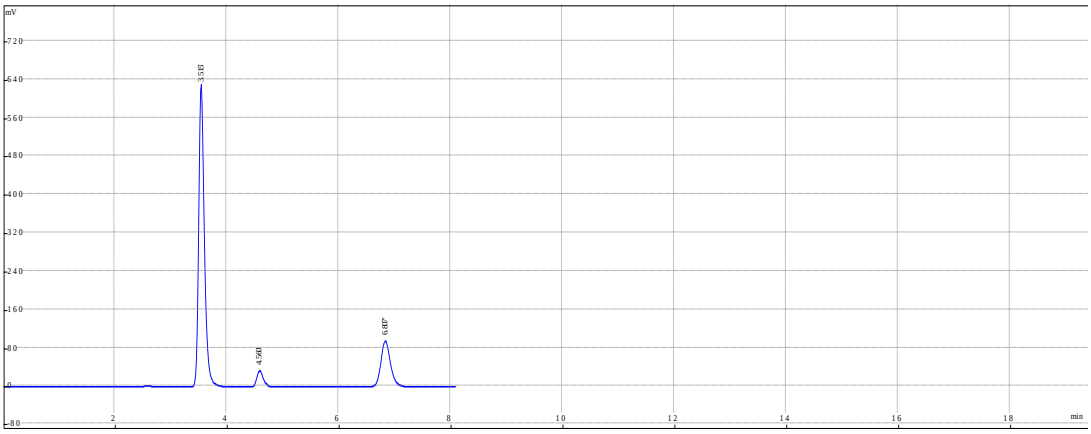

Figure No: 9 Chromatogram for Oxidative Degradation Study

Time	Area	Resolution	T. platinum	Asymmetry
6.807	1189700	0.00	7093	1.15

4] For Thermal degradation:

1mL stock solution of Dapagliflozin was kept in an oven which is maintained at 60^0c for about 30 min and later the volume was made up to mark with methanol then aliquot portion of above solution was diluted with mobile phase as a methanol (80%): water (20%) to get final concentration of about 100 µg/mL and 20 µL of sample solutions were injected and analyzed against control samples (lacking degradation treatment). Dapagliflozin is found to be degraded in Thermal condition. As shown in figure No. 10

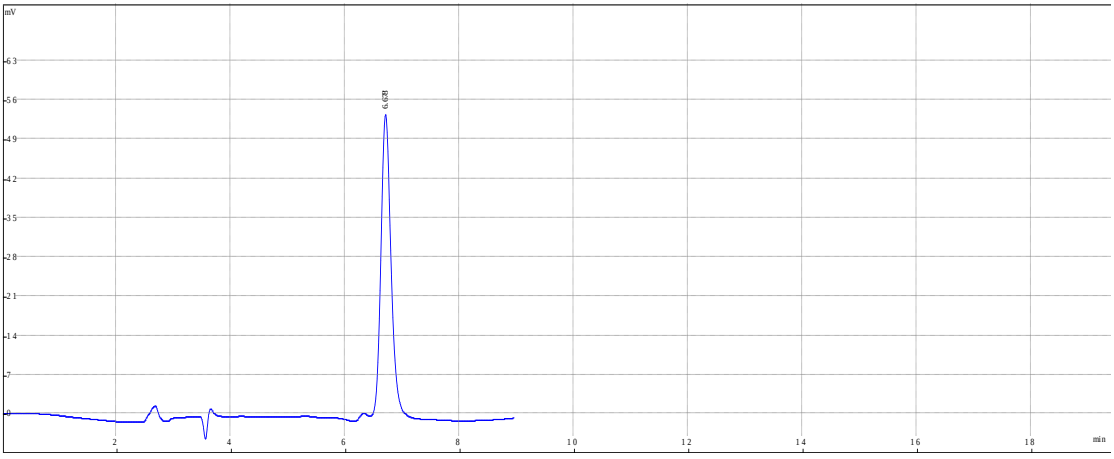

Figure No: 10 Chromatogram for Thermal Degradation Study

Time	Area	Resolution	T. platinum	Asymmetry
6.678	812187	0.00	6592	1.19

5] For Photolytic degradation:

1mg of Dapagliflozin was placed in Petri plate and exposed to sunlight@ for 30 min and later the volume was made up to mark with methanol then aliquot portion of above solution was diluted with mobile phase as a methanol (80%) : water (20%) to get final concentration of about 100µg/mL. and 20µL of sample solutions were injected and analyzed against control samples (lacking degradation treatment). Dapagliflozin is found to be degraded in Photolytic condition. As shown in **figure No.11**

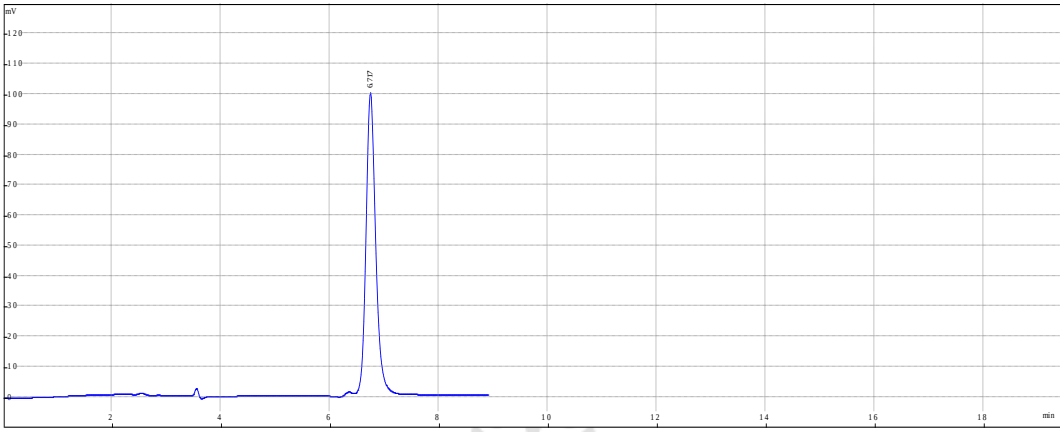

Figure No: 11 Chromatogram for Photolytic Degradation Study

Time	Area	Resolution	T. platinum	Asymmetry
6.717	1256410	0.00	6533	1.19

Table No: 10 Observations and Result of forced degradation study.

Stress condition	Exposure time	Condition	Peak area of degradant	% Degradation
Acidic	30 min	Refluxed at 60^0C	1025260	32.15
Basic	30 min	Refluxed at 60^0C	866132	27.23
Oxidation	12 hrs	Refluxed at 60^0C	1189700	37.31
Thermal	30 min	In oven	812187	25.47
Photolytic	30 min	Exposed to sunlight	1256410	39.41

RESULTS AND DISCUSSION

A new stability indicating HPLC method has been developed for estimation of Dapagliflozin in API and Tablet dosage form was rapid, accurate, precise, specific, sensitive and robust. From the above study, we can conclude that the Dapagliflozin was subjected to acid, alkali hydrolysis, and oxidation, thermal and photolytic degradation. Forxiga is more susceptible to photolytic degradation. From the above peak purity study, it was confirmed that the peak of degradation product and excipient was not interfering with the peak of the drug. Hence this method was used for the analysis of Dapagliflozin in API and tablet dosage form in quality control department for routine analysis.

Linearity of the developed method follows beer's law and was near to 0.999. It found to be linear in the range 10- 50µg/ml. % RSD was found to be less than 2 for precision. % Recoveries was found to be 99.92%.

CONCLUSION

In the present study, we have developed a new, rapid HPLC method and validated for different parameters (system suitability, linearity, accuracy, precision, ruggedness). Degradation is done under various conditions (acidic, alkaline, oxidation, photolytic and thermal). Degradation study indicates stability of the drug. Hence the method was successfully applied for the estimation of dapagliflozin in API and pharmaceutical dosage form.

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