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UPLC METHOD DEVELOPMENT AND VALIDATION FOR EDOXABAN ESTIMATION IN BULK AND PHARMACEUTICAL DOSAGE FORM

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ABSTRACT

In order to estimate Edoxaban in bulk and pharmaceutical formulations, this work designed and validated a reverse phase ultra-performance liquid chromatography method. Several technique elements, including mobile phase ratio and column type, were altered in statistically structured studies to evaluate the impact of these variables on the chromatographic separation of Edoxaban. Using ACN: Phosphate buffer (pH 3.0) (70:30), the separation was performed at ambient temperature under isocratic conditions at a flow rate of 0.2 mL/min on a BEH C18 (2.1 x 50mm, 1.7µm) Column and 5µm. The maximum absorbance 298 nm was measured by a PDA detector. The retention time was found to be 1.520mins whereas run time was 3mins only. In between the concentration range of 15-90 µg/mL, the calibration curve was linear. The new technique's sensitivity is demonstrated by the calculated LOQ of 0.45µg/mL and the measured LOD of 0.15µg/mL. The approach's robustness and ruggedness were confirmed by the %RSD being less than 2. For formulation analysis, the test percentage was 100.02. As a result, this technique was widely employed to analyse Edoxaban in bulk and pharmaceutical formulations.

KEYWORDS: Edoxaban, Method Development, Linearity, Accuracy and Precision.

INTRODUCTION

A direct oral anticoagulant, or blood thinner, edoxaban is used to treat and prevent potentially harmful blood clots.[1,2] Edoxaban has an inhibitory constant (Ki) of 0.561 nM and is a direct, specific, reversible, and competitive inhibitor of human factor Xa. On platelet surfaces, uncontrolled factor Xa and factor Va combine to create a prothrombinase complex during coagulation. Prothrombins are converted to thrombins by prothrombinases. Blood-soluble fibrinogens are converted by thrombins into insoluble fibrins, which are the primary constituents of blood clots.[3,4] Only a few analytical (UV, HPLC & HPTLC) and bioanalytical methods have been reported for the measurement of Edoxaban in bulk and pharmaceutical formulation, and no one has mentioned UPLC, which was more reliable and accurate than the reported literature. Edoxaban's chemical structure is shown in Fig. 1.[2]

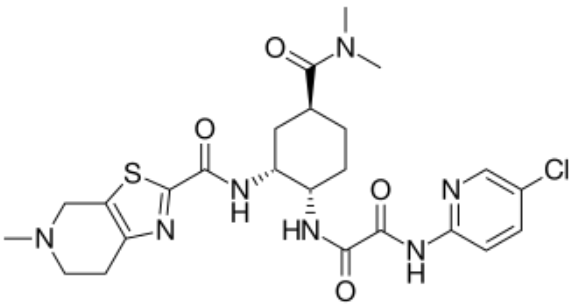

Fig. 1: The Chemical structure of Edoxaban.

MATEIALS AND METHODS

Chemicals and Reagents

The working standard drug Edoxaban (98% purity) was obtained from Dr. Reddy's Laboratories, Hyderabad, Telangana. The formulation dosage form having brand

name Supexa-OD 60 mg containing 60 mg of Edoxaban, was purchased from local Pharmacy. HPLC grade Methanol, Water and Acetonitrile, Potassium dihydrogen phosphate (KH_2PO_4) & Orthophosphoric acid (OPA) were purchased form Merk chemicals private limited, Mumbai.

Preparation of Mobile Phase

Acetonitrile: Phosphate buffer (pH 3.0) (70:30 v/v) was made. The mobile phase was sonicated for 15 minutes to remove dissolved gases, and then it was filtered through a 0.22 µm membrane filter before being used.

Preparation of standard drug solution

Edoxaban (60 mg) was transferred into a 10 ml volumetric flask after being carefully weighed. After that, it was fully dissolved using an ultra sonicator in 7mL of acetonitrile. A 0.22 µm membrane filter was used to filter the mixture, after the final volume in the volumetric flask was adjusted using the same solvent. The result was a standard stock solution of 6000 µg/mL. Using mobile phase as a diluent, the concentration (6000 to 600 to 60µg/mL) was prepared which was needed for method development and validation criteria.

Preparation of formulation solution

10 Edoxaban tablets (60 mg according to the label) were carefully weighed and pulverised into a fine powder. precisely weigh 60 mg equivalent Edoxaban tablet powder, was added to a 10 mL volumetric flask. About 7 mL of acetonitrile was added to the flask, and the mixture was sonicated for 15 minutes. The same diluent was used to adjust the volume once the solution had cooled to room temperature. The resultant solution was thoroughly mixed to produce a sample stock solution with a concentration of 6000 µg/mL. The sample stock solution was filtered using a 0.22 µm membrane filter, and the first few mL of the filtrate were thrown away. 1.0 mL of the filtered sample stock solution was carefully pipetted into a 10 mL volumetric flask and diluted to volume with the diluent to produce a working sample solution of 600 µg/mL. Additional dilutions were made from the working sample solution using the same diluent in order to achieve the required concentration 60 µg/mL for assay and validation testing.

METHOD DEVELOPMENT

Selection of Wavelength

To select a suitable wavelength, reference solutions containing 60µg/mL were scanned using the PDA detector. The best wavelength for the detection was determined to be the maximum wavelength that was obtained.

Table 1: Optimized Chromatographic Conditions.

Parameter	Condition
Mobile Phase	ACN: Phosphate buffer (pH 3.0) (70:30)
Column	BEH C18 (2.1 x 50mm, 1.7µm)
Flow Rate	0.2 ml/min
Wavelength	298nm
Injection Volume	5 µL
Temperature	Ambient
Run time	3 min

METHOD VALIDATION

The method was validated in terms of specificity, system suitability, LOD & LOQ, linearity, accuracy, precision, ruggedness, and robustness in compliance with the ICH requirements. Validation was carried out using duplicate injections of the sample and standard solutions into the column.[5]

RESULTS AND DISCUSSION

Method Development

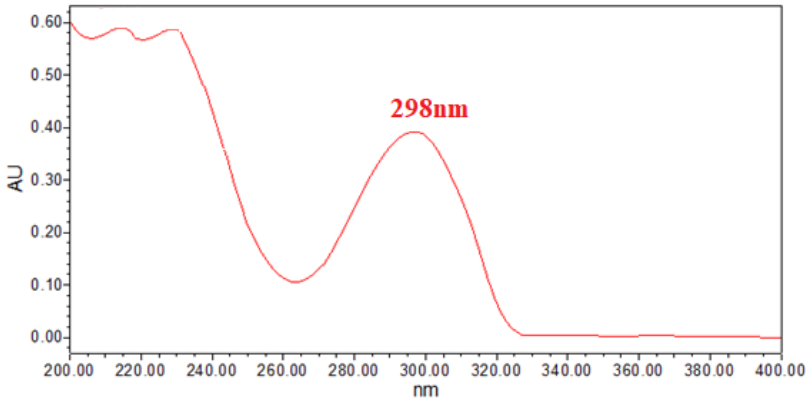

Fig. 2: UV Spectra of Edoxaban.

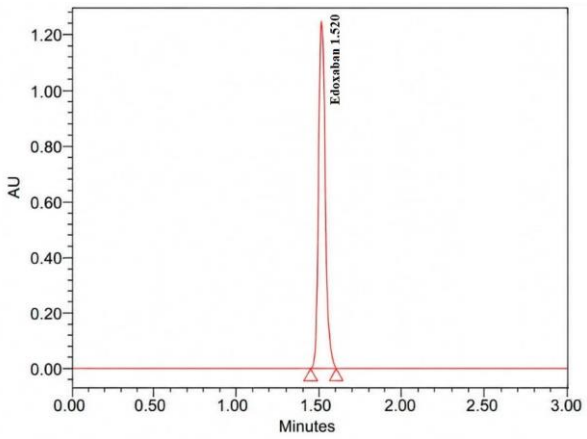

Fig. 3: Optimized Chromatogram of Edoxaban.

Table 2: Results for Optimized Chromatogram.

S. No.	Drug	Retention Time (min)	Theoretical Plates	Tailing Factor
1	Edoxaban	1.520	5189	1.38

METHOD VALIDATION

Specificity

No inference of diluent & Placebo

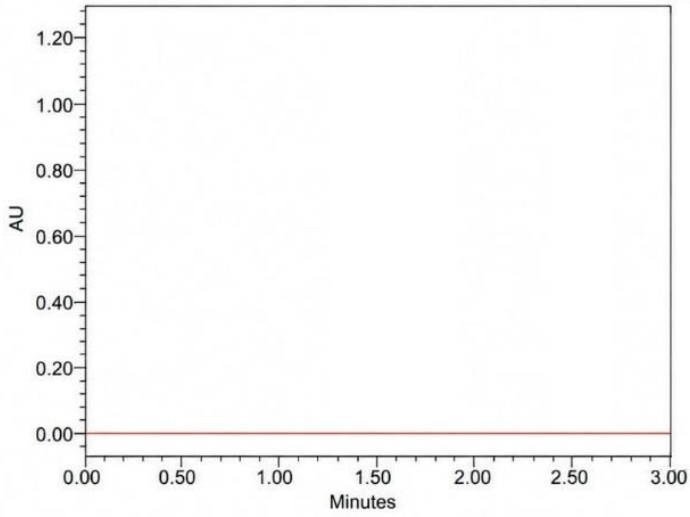

Fig. 4: Chromatogram of Blank.

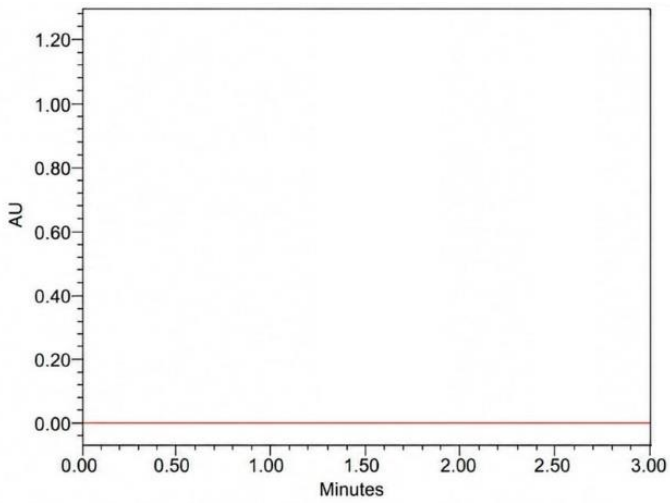

Fig. 5: Chromatogram of Placebo.

Linearity

Table 3: Results for Linearity.

S. No	Level	Edoxaban	
		Concentration in µg/mL	Peak Area
1	Level 1	15	113254
2	Level 2	30	216587
3	Level 3	45	319921
4	Level 4	60	423254
5	Level 5	75	526587
6	Level 6	90	629890

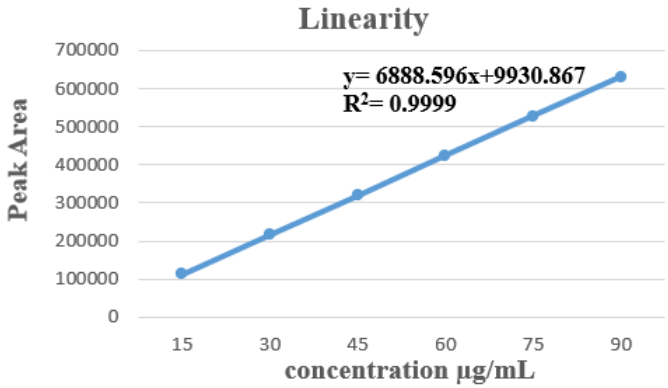

Fig. 6: Linearity graph for Edoxaban.

LOD & LOQ

Edoxaban 's LOD was found to be 0.15µg/mL, while its LOQ was determined to be 0.45µg/mL.

Precision

For Edoxaban, the system and method precision % RSD were found to be 0.37 and 0.19 respectively. The %RSD for both system and technique precision was within the acceptable range of less than 2. Consequently, the established procedure was considered to be precise.

Table 4: Results for Precision.

S. No.	Injection	System Precision		Method Precision	
		Retention Time	Peak Area	Retention Time	Peak Area
1	Injection-1	1.526	412345	1.523	413109
2	Injection-2	1.530	415987	1.520	411987
3	Injection-3	1.523	413210	1.523	413095
4	Injection-4	1.520	411876	1.525	413365
5	Injection-5	1.518	412567	1.518	411485
6	Injection-6	1.523	411998	1.520	411895
Mean		1.523333	412997.2	1.5215	412489.3
STD		0.004274	1539.488	0.0026	791.4376
%RSD		0.28	0.37	0.17	0.19

Accuracy

It was found that the recovery percentage ranged from 99.80 to 100.12% (Table 5). The percentage RSD was found to be within the acceptable limit for Edoxaban at 50%, 100%, and 150% spiking levels. With an acceptance limit of 98–102% and a percentage RSD of less than two, the results demonstrated the accuracy of the recommended technique.

Ruggedness (Intermediate Precision)

Ruggedness must be described by a percentage RSD of less than 2. Edoxaban's % RSD in the developed method was 0.19 (Table 6). Results that fall within the permitted range verify the process's ruggedness.

Robustness

The developed method's Edoxaban percentage change was confirmed to be within the acceptable range of less than 2. It was thus shown that the recommended method was suitable for the analysis of Edoxaban when the analytical conditions were slightly changed. This demonstrates that even slight modifications to the analytical conditions have no effect on the results (Table 7).

Table 5: Results for Accuracy.

Recovery Level	Concentration in µg/ml			Amount Found	% Recovery	% RSD
	Target	Spiked	Total			
50%	30	15	45	44.93	99.84	0.14
	30	15	45	45.03	100.06	
	30	15	45	44.91	99.80	
100%	30	30	60	59.94	99.90	0.11
	30	30	60	60.05	100.08	
	30	30	60	59.93	99.88	
150%	30	45	75	74.98	99.97	0.12
	30	45	75	74.92	99.89	
	30	45	75	75.09	100.12	

Table 6: Results for Ruggedness.

S. No.	Injection	Retention Time	Peak Area
1	Injection-1	1.530	415987
2	Injection-2	1.529	415258
3	Injection-3	1.535	419876
4	Injection-4	1.528	415119
5	Injection-5	1.521	413968
6	Injection-6	1.523	413549
Mean		1.527667	415626.2
STD		0.003606	812.9434
%RSD		0.19	0.19

Table 7: Results for Robustness

S. No.	Condition	Edoxaban		
		Retention Time	Peak Area	% Change
1	Standard	1.520	411598	--
2	+MP (75:25)	1.518	411126	0.11
3	-MP (65:35)	1.524	412168	0.13
4	Flow Rate 0.22ml/min	1.535	414259	0.64
5	Flow rate 0.18ml/min	1.528	412895	0.31
% RSD		0.45	0.94	

Assay

In formulation analysis, the test percentage for edoxaban was 100.02%. Consequently, it was shown that the method was suitable for routine analysis of Edoxaban in both formulation and bulk form.

Table 8: Results for Formulation.

S. No	Drug	Brand	Label Claim	Peak Area	Amount Found	% Assay
1	Edoxaban	Supexa-OD 60	60 mg	411687	60.01mg	100.02

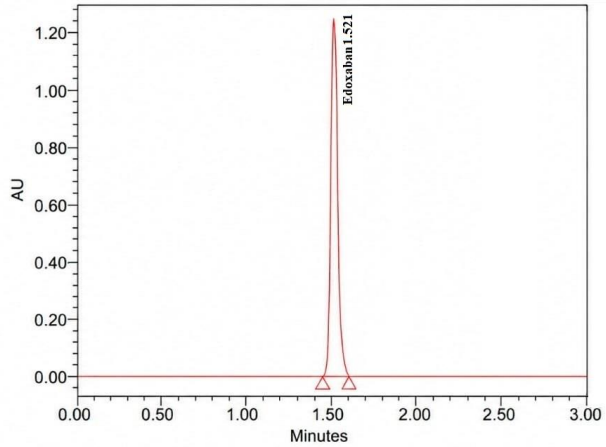

Fig. 7: Chromatogram of Formulation.

CONCLUSION

Before this study began, there was little information available in the literature about the HPLC measurement of Edoxaban in pharmaceutical formulations. The author has created a sensitive, precise, and accurate RP-UPLC method for measuring Edoxaban in bulk and pharmaceutical formulations. After validation, it was demonstrated that the suggested RP-UPLC method for the Edoxaban test was appropriate for routine quantitative analysis. The UPLC approach eliminated the need for laborious extraction and saved time when preparing the standard and sample. The new method's extraordinarily high precision is demonstrated by the low standard deviation statistics. It was found that the values for linearity, accuracy, specificity, and precision fell within acceptable bounds. The absence of additional peaks in the chromatogram demonstrated that the tablet's common excipients did not conflict with one another. The developed RP-UPLC approach is therefore demonstrated to be simple, linear, accurate, sensitive, and repeatable. For routine quality monitoring of Edoxaban in pharmaceutical formulations and bulk, the developed method is therefore easy to use and has a quick analytical time. The provided results show that the recommended method has good accuracy and precision.

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