Simultaneous Estimation of Aspirin and Atorvastatin Calcium in Bulk and Pharmaceutical Formulation Using by UV-Visible Spectroscopy

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***Abstract*—A simple UV-Visible Spectrophotometric method was developed and validated for the Simultaneous estimation of Atorvastatin and Aspirin in bulk and dosage forms. Atorvastatin and Aspirin stock solutions of 20 µg/mL were prepared in 0.1N sodium hydroxide as diluent and scanned in UV- region for the determination of λmax and was 243nm and 276nm respectively. Linearity was established over the concentration range of 5-35 µg/ml for Atorvastatin and Aspirin with correlation coefficient (R^2) value 0.9958 and 0.9955 respectively. The method was validated according to ICH guidelines for validation parameters like accuracy, precision, LOD, LOQ and assay parameters. All the parameters validated were to be within limits. The method was successfully applied for quantitative estimation of Atorvastatin calcium and Aspirin in capsules by UV method.**

***Index Terms*—Atorvastatin calcium, Aspirin, Simultaneous estimation, UV spectroscopy, Validation,**

I. INTRODUCTION

Atorvastatin Calcium is: [(βR, ds)-2-(4-flouorophenyl)-β, δ-di hydroxy-5- (1-methylethyl) -3-phenyl-4 [(phenyl amino) carbonyl]-1 heptenoic acid calcium salt (ATV). It is statin used for lowering blood cholesterol. It is a selective competitive inhibitor of the enzyme HMG CoA reductase, which catalyzes the conversion of HMG-CoA to mevalonate, an important rate limiting step in cholesterol biosynthesis. Aspirin (ASP) is chemically known as acetyl salicylic acid and is used as non-steroidal anti-inflammatory and analgesic drug

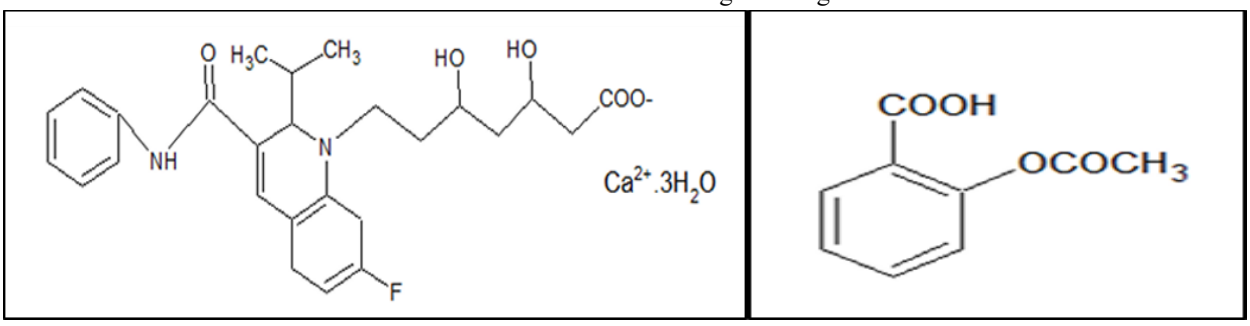

Figure -01 Structure of Atorvastatin calcium and Aspirin

Both Atorvastatin calcium and Aspirin are freely soluble in NaOH, slightly soluble in ethanol and very slightly soluble in distilled water. As per literature UV spectrophotometric methods are available for simultaneous estimation of Atorvastatin calcium and Aspirin in marketed formulations. To the best of our knowledge there is spectrophotometric method reported for determination of ATV and ASP simultaneously. Therefore, an attempt has been made to develop a simultaneous equation method by UV spectrophotometric method for analysis of these two drugs using different dosage forms available in market.

II. MATERIAL AND METHODS

A. Instrument used- A Lab India UV- Visible double beam spectrophotometer with spectral band width of

1.8 nm, wavelength accuracy of ± 2 nm and matched quartz cells of 10 mm optical path length was used for all spectral and absorbance measurements.

Reagents and chemicals: The Standard of Atorvastatin calcium and Aspirin was taken and the dosage form Aztor ASP 75, Ecosprin AV 75, Atorec ASP, Modlip ASG 75, Atorlip-ASP 10 were procured from market and utilized for the study. All chemicals and reagent used were of analytical grade

Preparation of blank solution - 0.1 N NaOH is used as blank solution.

B. Preparation of Standard Stock Solution and Calibration curve

Pure Drug: Accurately weighed and transferred 25mg pure drug of ATR and ASP were dissolved in the 0.1N NaOH in a two different 10ml volumetric flask and sonicated for 10min and made up the volume to mark with the diluent to make a conc of 1mg/mL from which 10mL of the solution was taken and diluted to 100mL to obtain a conc. range of 100µg/mL. From the resultant stock solution seven working standard solution with concentration 5, 10, 15, 20, 25, 30, 35, µg/ml of Atorvastatin calcium and 5, 10, 15, 20, 25, 30 and 35 µg/ml of Aspirin was taken respectivley. The absorbance of resulting solution were measured at their respective λmax and plotted a calibration curve to get linearity and regression equation.

Valuation of absorption maxima

The two solutions of ATR and ASP were scanned separately in the range of 200-400 nm to determine respective wavelength of maximum absorption. ATR and ASP showed absorbance maxima at 243 nm (λ1) and 296 nm (λ2) respectively.

C. Marketed formulation

Pooled Twenty capsules of respective formulation procured were accurately weighed, and contents were removed. Average weight of the content per capsule was calculated. The contents of a capsule were reduced to fine powder. A quantity of capsule powder equivalent to Label claim of Atorvastatin calcium of Aspirin accurately weighted and taken in a 100 ml of volumetric flask containing 10ml 0.1N NaOH. The solution was sonicate for 30min with occasional shaking and allowed to cool to room temp and was filtered through Whatmann filter paper no.40.

From the prepared stock solution 20mL and 2.7mL were transferred separately into 100mL volumetric flask and made up to the volume with diluent to obtain the conc. of 20 µg/mL ATR and ASP respectively. The absorbance of resulting solutions was measured at 243 nm and 296 nm. Values were substituted in the respective formula to obtain concentrations.

D. Simultaneous Equation Method

The Simultaneous Equation Method of analysis based on the absorption of the drugs Atorvastatin calcium and Aspirin at their λmax. Two wavelengths selected for the development of Simultaneous Equation were 243 nm (λ1) and 296 nm (λ2). Absorptivity of both the drugs at both the wavelengths were determined. The equations obtained for the estimation of concentration were,

$Cx = A2ay1 - A1ay2 / ax2ay1 - ax1ay2$

$Cy = A1ax2 - A2ax1 / ax2ay1 - ax1ay2$

Where,

ax1- absorptivity at 243nm of ATR, ax2- absorptivity at 296nm of ATV

ay1- absorptivity at 243nm of ASP, ay2- absorptivity at 296nm of ASP

A1 = Absorptivity of ATR at 243 nm, A2= Absorptivity of ASP at 296 nm

CX and CY are concentration of ATR and ASP in sample solution

E. Validation Parameters

Linearity: The linearity of measurement was evaluated by analyzing different concentration of the standard solution of ATR and ASP (Figure 1 & 2). For simultaneous equation method the Beer- Lambert's concentration range was found to be for 5-35 µg/ml for ATR and ASP were selected and determined for regression analysis.

Precision – The precision analysis was performed by measuring the 100% test conc. in six replicates and determined for %RSD for the given samples of ATR and ASP in standard solution respectively. Both reproducibility and repeatability of precision were studied.

Accuracy: To ascertain the accuracy of the proposed methods, recovery studies were carried at three different levels 80%, 100% and 120% as per ICH guidelines

Limit of Detection (LOD) and Limit of Quantization (LOQ): The LOD and LOQ of ATR and ASP by proposed methods were determined using calibration standards. LOD and LOQ were calculated as $3.3\sigma/S$ and $10\sigma/S$ respectively, where S is the slope of the calibration curve and σ is the standard deviation of response.

III. RESULTS AND DISCUSSION

Studies on calibration curves, precision, and recovery were conducted under the stated experimental circumstances. With good correlation coefficients of 0.9958 and 0.9955, respectively, in the concentration range of 5–35 µg/ml for ATR and ASP. Table 1 displays the findings from recovery investigations. The findings of the analysis of commercial formulations are shown in Table 2. Given that the results are nearly 100% accurate and have a low standard deviation, the data clearly shows accuracy and reproducibility. The suggested techniques are quick, easy, accurate, precise, and affordable. For routine analysis of ATR and ASP in capsule formulation, these can therefore be utilized.

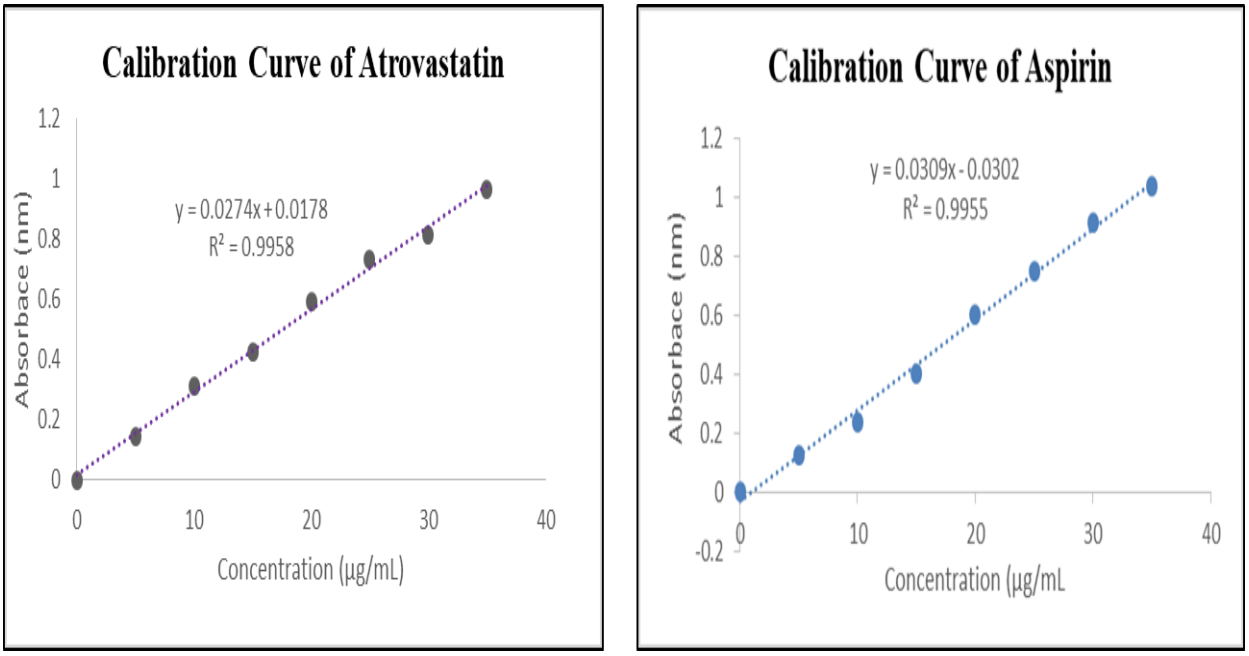

Figure-02 Calibration curve for ATR and ASP

Table-01 Data acquired for validation parameters

S.NO	Parameters		Result for ATR	Result for ASP
01	Absorption maxima (λ max)		243nm	296nm
02	Beer's law limit (µg/ml)		5-35	5-35
03	Regression equation		y = 0.0274x + 0.0178	y = 0.0309x - 0.0302
04	Correlation coefficient (R^2)		0.9958	0.9955
05	Accuracy (%Recovery)		98.23	98.09
06	Precision Intraday RSD		0.268	0.176
07	Precision Assay		100.81	103.15
08	LOD		2.14	3.23
09	LOQ		6.50	9.77
10	Assay	Aztor	97.97	102.28
		Midilop	100.67	95.63
		Atorec	99.72	100.55
		Ecosprin	101.61	97.50
		Atrolip	96.35	100.21

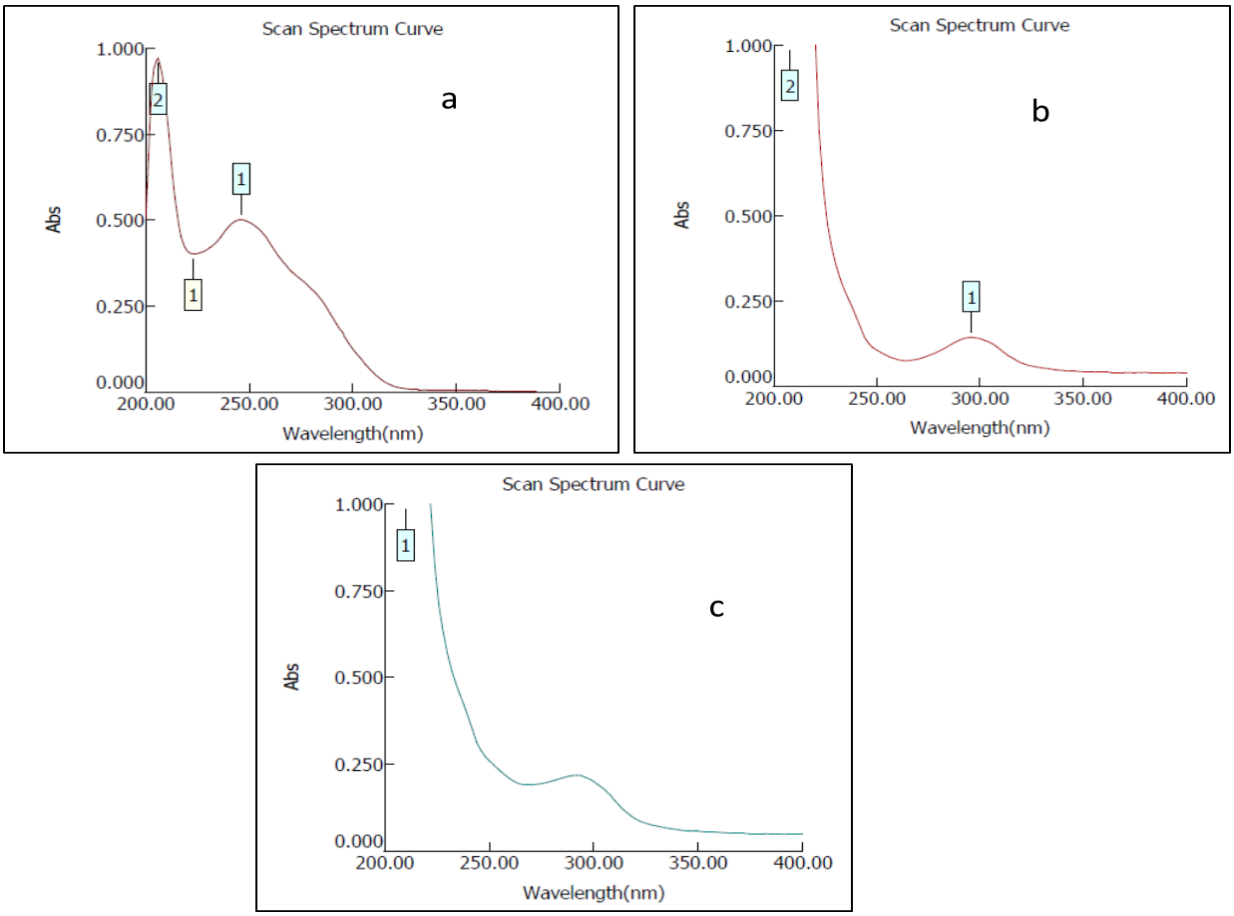

Figure-03 a.UV Spectrum of ATR b. UV Spectrum of ASP c. UV Spectrum of Mixture

Table- 02 Accuracy data

S.NO		Amt added	Abs	Intercept	Slope	y-c	y-c/m	% recovery
	Regression equation		y = 0.0274x + 0.0178					
01	ATR	36	0.985	0.0178	0.0274	0.9672	35.30	98.05
02		40	1.101	0.0178	0.0274	1.0832	39.53	98.83
03		44	1.197	0.0178	0.0274	1.1792	43.04	97.81
	Regression equation		y = 0.0309x - 0.0302					
01	ASP	36	1.056	0.0302	0.0309	1.0862	35.15	97.64
02		40	1.187	0.0302	0.0309	1.2172	39.39	98.48
03		44	1.304	0.0302	0.0309	1.3342	43.18	98.13

IV. CONCLUSION

In the preceding analysis, concentration was estimated using the earlier equations, and percent purity was estimated using the simultaneous equation method. The findings achieved using the suggested approach were similar to the label claims of both medicines. Linearity for Aspirin and Atorvastatin Calcium ranged from 5 to 35 µg ml-1 for ATR and ASP. The low standard deviation suggests that the approaches are accurate. The suggested approach's accuracy and repeatability were verified using recovery trials utilizing the usual addition method. All of the formulations examined were determined to be accurate, simple, and fast for simultaneous estimate of marketed formulations. Thus, the suggested methods

for simultaneous assessment of Aspirin and Atorvastatin Calcium in capsule dosage forms were discovered to be quick, sensitive, easy, and accurate. Therefore, the method can be useful in routine quality control of these drugs

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