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Development and Validation of RP-HPLC Method for Simultaneous Determination of Aspirin and Omeprazole

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ABSTRACT

In this study, reverse phase high performance liquid chromatographic method have been developed and validated for the simultaneous determination of aspirin and omeprazole. The chromatographic separation was achieved in a Zorbax Eclipse XDB- C18 (4.6 × 250 mm × 5 µ) as a stationary phase Acetonitrile: Water (50:50, v/v) as mobile phase at a flow rate of 1 ml/min. UV detection was performed at 293 nm. The retention time of aspirin and omeprazole was found to be 3.260 and 1.787 min respectively. The results of analysis were validated statistically and by recovery studies. Linearity, accuracy and precision were acceptable in the ranges (2-14 µg/ml) for aspirin and (2-18 µg/ml) for omeprazole. The % recovery for aspirin and omeprazole was 99.79 and 99.61, respectively. The results of the studies showed that the proposed Reverse Phase High Performance Liquid Chromatography (RP-HPLC) method was simple, rapid, precise and accurate, which can be used for the routine determination of aspirin and omeprazole.

Keywords: Aspirin, Omeprazole, Liquid chromatography, Validation

INTRODUCTION

Aspirin (ASP) was Nonsteroidal anti-inflammatory, antirheumatic, antithrombic and chemically it was 2-Acetoxy benzoic acid. Chemical Structure of Aspirin was given in Figure 1, molecular formula was $C_9H_8O_4$, molecular weight is 180.16 g/mol [1].
Omeprazole (OME) was proton pump inhibitors chemically it was 5-Methoxy-2-[[(4-methoxy-3,5-dimethyl-2-pyridinyl)-methy]sulfinyl]-1H-benzimidazole, chemical structure of omeprazole was given in Figure 2, molecular formula was $C_{17}H_{19}N_3O_3S$, molecular weight is 345.42 g/mol [2].

Literature survey revealed that there are various methods have been reported for estimation of ASP and OME by High Performance Liquid Chromatography (HPLC) method [3-7], UV-spectroscopic methods [8-10], Liquid Chromatography-Mass Spectrometry (LC-MS) [11-13] and High-performance Thin-Layer Chromatography (HPTLC) method [14] individually and in combined dosage form with other drugs. But no single method is available in combination by using this mobile phase. The present work therefore emphasizes on the quantitative estimation of ASP and OMP in synthetic mixture by HPLC. The proposed method was validated as per the International Conference on Harmonization (ICH) analytical method validation guidelines [15].

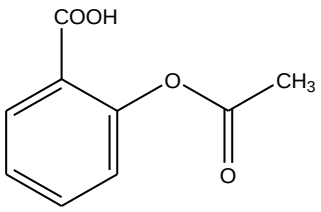

Figure 1: Aspirin

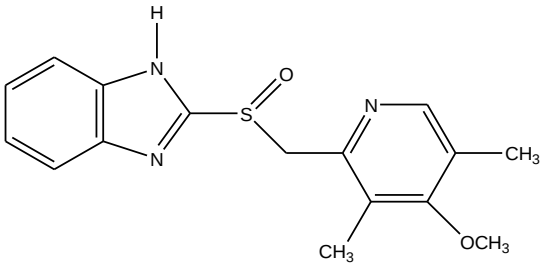

Figure 2: Omeprazole

A new combination of aspirin and omeprazole was approved as an initial once a day therapy for Ischemic stroke-prophylaxis, gastric ulcer prophylaxis and cardiovascular disease. Literature survey reveals that there was no RP-HPLC method available for the determination of these analytes in combination; therefore the aim of this paper was to develop a specific, precise and accurate chromatographic method that could be applied in quality control for the simultaneous determination of aspirin and omeprazole.

MATERIALS AND METHODS

Reagents and materials

Pure samples of aspirin were purchased from Swapnroop Pharmaceutical, Aurangabad and Omeprazole was purchased from Zydus Cadila, Gujarat. Acetonitrile of HPLC grade was purchased from Merck (India) Ltd., Mumbai. Mille Q water was used throughout the process.

Chromatographic conditions

The isocratic mobile phase consisted of water: acetonitrile in the ratio of 50:50 (v/v), flowing through the column at a constant flow rate of 1 ml/min. Zorbax Eclipse XDB-C18 column (4.6 × 250 mm × 5 µ) was used as the stationary phase. By considering the chromatographic parameter, sensitivity and selectivity of method for two drugs, 293 nm was selected as the detection wavelength for UV detector. The HPLC system was (Agilent 1220 LC) [16].

Preparation of standard stock solution

Accurately, about 10 mg of standard ASP and OME was weighed and transferred to separate 10 ml volumetric flasks. The drugs were dissolved in ethanol then volume made up to the mark with same solvent to obtain standard stock solution of each drug of concentration 1000 µg/ml.

Preparation of standard working solution

From standard stock solution appropriate aliquot portion 1 ml is transferred to 10 ml volumetric flask and diluted with ethanol to obtain the concentration 100 µg/ml to this appropriate aliquot portion 0.81 ml ASP and 0.4 ml OME was transferred to 10 ml volumetric flask and diluted with ethanol to obtain the concentration 8.1 µg/ml of ASP and 4 µg/ml of OME. A volume of 20 µl of solution was injected with the help of Hamilton Syringe. All measurements were repeated three times for each concentration and from the peak area. Chromatogram is shown in Figure 3.

Linearity and range

It was performed using different test concentrations. Response was Linear in the range of 2-14 µg/ml for ASP and 2-18 µg/ml OME. A calibration curves were plotted between the mean peak areas *vs.* respective concentrations calibration curve is shown in Figures 3 and 4 for ASP and OME respectively. The corresponding linear regression equation is y=4E+06x+740673 with square of correlation coefficient R^2 of 0.9985 for aspirin and y=301514x+5E+06 with square of correlation coefficient R^2 of 0.9992 for Omeprazole, respectively.

Specificity

The specificity of the method was determined by checking the interference with the components. The Figure 5 showed typical chromatogram obtained from analysis of standard solution using the proposed method. The retention time observed 3.260 min for aspirin and 1.787 min for Omeprazole permits a rapid assay, which was important for routine analysis.

Recovery study

Known amount of standard ASP and OME was added to pre analyzed sample (8, 10 and 12 µg/ml) and subjected them to the proposed HPLC method.

Precision

System repeatability was done by repeating the assay three times of the same concentration after every 2 h on the same day for intraday precision. Interday precision was carried out by performing the assay sample sets after 24 h and 48 h. Sensitivity of the proposed method was estimated in terms of Limit of Detection (LOD) and Limit of Quantitation (LOQ). LOD=3.3 SD/S and LOQ=10 SD/S, where SD was the residual standard deviation and S was the slope of the line. LOD was found to be 0.67 ng/ml and LOQ was found to be 1.94 ng/ml for ASP and LOD was found to be 0.43 ng/ml and LOQ was found to be 1.25 ng/ml for OME.

Ruggedness

From stock solution, sample solution of ASP and OME (10 µg/ml) was prepared and analyzed by two different analysts using similar operational and environmental conditions. Peak area was measured for same concentration solutions (Table 1).

RESULTS AND DISCUSSION

Table 1: Summary of validation parameter ASP and OME

Parameters	ASP	OME
Linearity range [µg/ml]	2-14	2-18
Regression equation [Y=mX+C]	4E+06X+74067	30151X+5E+06
Recovery [% RSD]	0.33115	0.569859
Precision [% RSD]		
Intra-day	0.372951	0.421925
Inter-day	0.229772	0.675527
Ruggedness [% RSD]	0.609453	1.153796
LOD	0.67	0.43
LOQ	1.94	1.25

[Mean(n)=3, % RSD=Relative Standard Deviation]

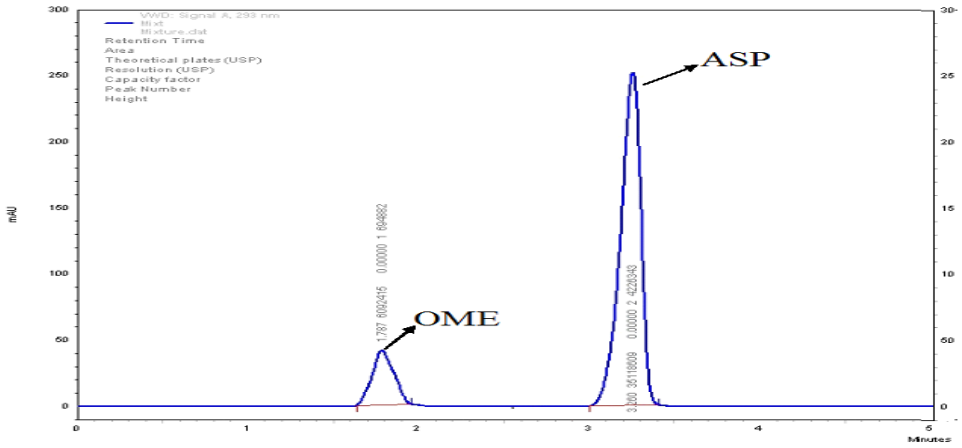

Figure 3: Chromatogram of aspirin and omeprazole

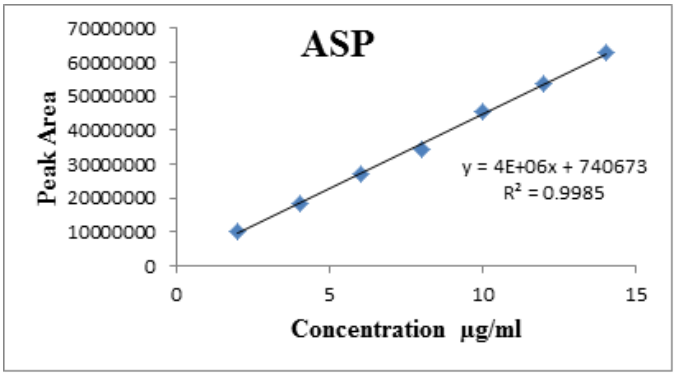

Figure 4: Calibration curve of ASP

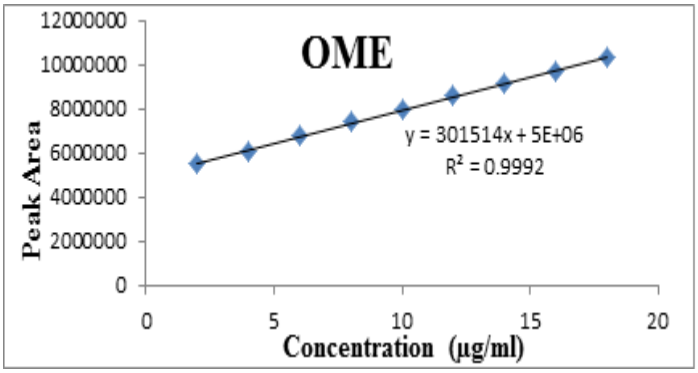

Figure 5: Calibration curve of OME

CONCLUSION

The proposed methods have proved to be simple, rapid, precise, accurate sensitive and economical and are suitable for simultaneous quantification of aspirin and omeprazole.

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REFERENCES

[1] Indian Pharmacopeia 6th edition, Government of India, Ministry of Health and Family Welfare, Ghaziabad, New Delhi. The Indian pharmacopeia commission publisher, **2010**, 2, 842-843.
[2] Indian Pharmacopoeia 6th edition, Government of India, Ministry of Health and Family Welfare, Ghaziabad, New Delhi, The Indian pharmacopoeia commission publisher, **2010**, 3, 1813-1814.
[3] S. Rajput, S. Fanse, *J. Adv. Pharm. Edu. Res*., **2015**, 5(2), 87-93.
[4] F.J. Chodvadiya, K.C. Thula, D.G. Maheshwari, *Int. J. Recent Sci. Res*., **2015**, 6(4), 3385-3390.
[5] D. Jain, N. Jain, J. Verma, *Int. J. Pharm. Sci. Drug Res*., **2012**, 4(3), 218-221.
[6] D. Godavariya, B. Prajapati, P. Marolia, S.A. Shah, *Int. Res. J. Pharm.*, **2012**, 3(8), 173-175.
[7] C. Solanki, N. Patel, *Int. J. Pharm. Bio. Sci*., **2012**, 3(3), 577-585.
[8] G. Kumaraswamy, L. Repeudi, K. Vijayaprakash, *Int. J. Pharm. Chem. Anal*., **2016**, 3(2), 90-98.
[9] B.B. Patel, B.B. Shah, K.N. Gohil, P.M. Patel, *Int. J. Res. Pharm. Sci*., **2012**, 2(2), 115-122.
[10] A.J. Purkar, A.R. Balap, S.B. Jadhav, P.D. Chaudhari, *Int. J. Pharm. Chem. Sci*., **2012**, 1(3), 1008-1012.
[11] E.R. Wickremsinhe, T. Ye, K.J. Ruterbories, E.M. Verburg, G.J. Weerakkody, A. Kurihara, N.A. Farid, *Drug. Metab. Dispos*., **2007**, 35, 917-921.
[12] N.A. Farid, M. McIntosh, F. Garofolo, E. Wong, A. Shwajch, M. Kennedy, M. Young, P. Sarkar, K. Kawabata, M. Takahashi, H. Pang, *Rapid Commun. Mass. Spectrochem.*, **2007**, 21, 169-179.
[13] O. Lukram, M. Zarapkar, C.K. Jha, S. Parmar, K.S. Tomar, A. Hande, *Drug. Test. Anal*., **2012**, 4, 58-166.
[14] T.C. Borole, R. Mehendre, M.C. Damle, K.G. Bothara, *J. Chem. Pharm. Res*., **2010**, 2, 907-913.
[15] ICH-Guidelines Q2 (R1), Validation of Analytical Procedures: Text and Methodology, **2005**, 1-13.
[16] Instruction manual model HPLC 2080 pump, Jasko Corporation.