International Journal of Research in Pharmacology & Pharmacotherapeutics (IJRPP)

ISSN:2278-2648

IJRPP |Volume 12 | Issue 2 | Apr - Jun – 2023
www.ijrpp.com

Research article Pharmaceutical Analysis

Spectrophotometric Determination of Aspirin by Using FC Reagent

A. Raja Reddy[1*], N.Divya[2], P.Pravalika[3], S. Hashika Keerthana[4], R.Kiran[5]

[1]*Associate Professor, Department of Pharmaceutical Analysis, CMR College of Pharmacy, Hyderabad,*
[2]*M. Pharm student, Department of Pharmaceutical Analysis, CMR College of Pharmacy, Hyderabad*
[3]*Associate Professor, Department of Pharmaceutical Analysis, CMR College of Pharmacy, Hyderabad*
[4]*M. Pharm student, Department of Pharmaceutical Analysis, CMR College of Pharmacy, Hyderabad*
[5]*Assistant Professor, Department of Pharmaceutical Analysis, CMR College of Pharmacy, Hyderabad*

***Corresponding author: A. Raja Reddy**
Published on: 28.04.2023

ABSTRACT

Aspirin is an antiplatelet agent therefore it is made to develop a new simple, sensitive and cost-effective UV spectrophotometric technique for the estimating aspirin using Folin Ciocalteu has been devised. The approach is based on formation of blue colored Chromagen as a result of Folin-Ciocalteu reagent reacts with the phenolic groups in aspirin. The spectrum was produced, and the maximum absorbance was found at 750 nm, with the beers law obeying across the concentration range 2-20 µg/ml with correlation coefficient found to be 0.9997. The method was validated for various parameters such as accuracy, precision robustness. The %RSD of precision was found to be 0.94%. The limit of detection and quantification value are 0.43 and 1.31 µg/ml respectively. The results of the proposed method were statistically validated as per ICH Q2 R1 guidelines and results were found to be satisfactory.

Keywords: Aspirin, Folin-Ciocalteu reagent, UV Spectrophotometric method, Validation.

INTRODUCTION

Acetylsalicylic acid is commonly referred to as aspirin. The British National Formulary classifies aspirin as a non-steroidal anti-inflammatory medicine a “anti-platelet” drug and a “non-opioid analgesic”[1] Salicylates were extracted from willow bark and were employed as an analgesic by Hippocrates, and their antipyretic properties have been known for almost 200 years. while aspirin was developed in the late 1890s and has been used to treat a range of inflammatory disorders, its antiplatelet action was not discovered until nearly 70years later.[2] In 1897, scientist Felix Hoffmann German company Bayer discovered aspirin for the first time. The name was derived from a combination of acetyl and Spiraea (Latin name for meadowsweet).[3]Aspirin acts as an antipyretic and analgesic. It works as an anti-inflammatory and antirheumatic drug by inhibiting prostaglandin. It also appears to generate analgesia via both peripheral and CNS effects. Aspirin will also prevent platelet aggregation and vasoconstriction by irreversibly inhibiting the cyclooxygenase-1 and thus inhibits the thrombooxygenase A_2.[4]Aspirin is readily absorbed in the upper gastrointestinal tract and inhibits platelet activity within 60mins.In the small intestine, aspirin absorption is pH sensitive. For the same pH range, small intestine absorption is greater than stomach absorption. Acetyl salicylic acids rapid metabolism produces salicylic acid, an active metabolite with residual anti-inflammatory activity[5].Literature survey reveals that numerous spectrophotometric methods are defined for determining aspirin

in combination with other drugs. since no method for single drug analysis has been published. As a result, an attempt has been made to establish simple, sensitive and validated method for estimation of drug in pharmaceutical formulation.[6-14]

ACETYL SALICYLIC ACID(ASPIRIN)

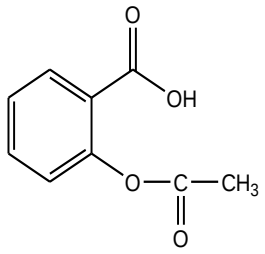

Fig 1: Structure of aspirin

MATERIALS AND METHODS

Instrumentation

PG Instrument (T60 UV-Visible spectrophotometer), UV win software using glass cuvettes of 10 mm path length, weighing balance (Pioneer OHAIUS (Item PA214C)).

Materials and Reagents

All chemicals and reagents used were of analytical grade. Folin-Ciocalteu (Merck, Mumbai) Sodium hydroxide (Hetero drugs Pvt Ltd, Hyd) were of analytical reagent grade. The Pharmaceutical grade pure drug was kindly provided by CMR College of pharmacy. Marketed drug obtained from local pharmacy.

Preparation of Standard Solution

A stock solution of aspirin is prepared by dissolving 100 mg pure drug in 100 ml distilled water in a volumetric flask Pipette out 1 ml of the above stock solution and is diluted to 10 ml. From the above solution pipette out 1 ml and makeup to 10 ml with solution. Folin Ciocalteu reagent solution (2N) is used as such. Sodium hydroxide (2N) is prepared by dissolving 4g of NaoH in 100 ml distilled water.

Determination of absorption maxima of Aspirin solution

The aspirin solution equivalent to 10 µg/ml was mixed with 2 ml of sodium hydroxide solution and 2 ml of Folin Ciocalteu reagent in 10 ml volumetric flask. After 15 mins the volume was made upto the mark with sodium hydroxide solution and it is mixed thoroughly. A blank solution was prepared in the same way in absence of aspirin. The solution is scanned in the range 400-800nm against reagent blank and Maximum absorption was observed at 750 nm.

Preparation of sample solution of Aspirin

About 10 tablets were weighed and crushed into fine powder. Powder weighed equivalent to 10 mg of Aspirin was transferred into a 100 ml volumetric flask and made upto 100 ml with sodium hydroxide and thoroughly agitated for 5 minutes. The content was kept aside for 5 mins, and it is filtered. The filtrate solution was properly diluted with solvent to obtain a required concentration of drug used for the analysis.

Validation Parameters

The method was validated according to ICH Q2B guidelines for validation of analytical procedures in order to determine the accuracy, linearity, precision, robustness of solution.

Linearity

Different aliquots of the working standard of aspirin solution ranging from 2-20 µg/ml was transferred into a series of volumetric flask and the total volume is made upto 10 ml with solvent. To each flask 2 ml of Folin Ciocalteu reagent solution was added by means of micro burette. The flask contents were mixed and kept at room temperature for 15 mins. The volume is made upto the mark with solvent and absorbance of each solution is measured at 730 nm against the blank. The linearity data is shown in table no 1.

Precision

The reproducibility of the proposed method was determined by evaluating the mid concentration of the standard solution (8 µg/ml) at different time intervals so measure the six replicate absorbance measurements intra and interday precision, %RSD was calculated, the data is shown in table no 3.

Accuracy

To ascertain the accuracy of the proposed methods, recovery studies were carried out at three different levels (80%, 100%, 120%). The accuracy was performed by spiking 0.8 ml of standard solution(8µg/ml) with 14.4 ml, 16 ml, 17.6 ml of working standard solution. They were made-up to mark with solvent and samples are carried out in triplicates. The accuracy data is shown in table no 7.

Robustness

The robustness of analytical procedure is the measure of its capacity to remain unaffected by small but deliberate variations in method parameters and provides an indication of its reliability during normal usage. The robustness of data was shown in table no 6.

RESULT AND DISCUSSION

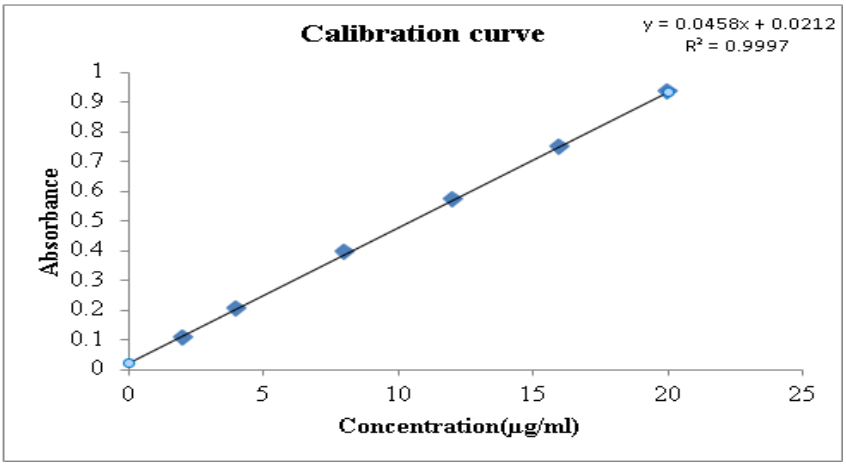

Fig 2: Calibration curve of aspirin

Table 1: Linearity of aspirin

Concentration (µg/ml)	Absorbance
2	0.106
4	0.204
8	0.398
12	0.572
16	0.75
20	0.935

Table 2: System suitability parameters of aspirin

S.no.	Optical characters	Results
1	λmax	750 nm
2	Beers law limit (µg/ml)	2-20 µg/ml
3	Slope	0.0453
4	Correlation Coefficient	0.9997

Table 3: Intraday Precision of aspirin

Concentration (µg/ml)	Absorbance
8	0.396
8	0.397
8	0.397
8	0.398
8	0.399
8	0.401
Mean	0.398
Standard deviation	0.0017
%RSD	0.4494

Table 4: Interday precision of aspirin

Concentration (µg/ml)	Absorbance (Day 1)	Absorbance (Day 2)
8	0.401	0.403
8	0.403	0.405

8	0.403	0.404
8	0.403	0.405
8	0.404	0.406
Mean	0.402	0.404
Standard deviation	0.0010	0.0010
%RSD	0.271	0.281

Table 5: LOD and LOQ of aspirin

Parameters	Aspirin (µg/ml)
LOD	0.43
LOQ	1.31

Table 6: Robustness of aspirin

S.no.	Wavelength	Absorbance
1	748 nm	0.347
2	750 nm	0.395
3	752 nm	0.404

Table 7: Accuracy of aspirin

S.no.	Sample level (%)	Amount taken (µg/ml)	Amount added (µg/ml)	Amount recovered (µg/ml)	Mean	% Recovery
1	80	8	14.4	7.9	7.9	99%
	80	8	14.4	8.0		
	80	8	14.4	8.0		
2	100	8	16	8.2	8.3	103%
	100	8	16	8.3		
	100	8	16	8.4		
3	120	8	17.6	8	8.3	104%
	120	8	17.6	8		
	120	8	17.6	9		

Table 8: Assay of aspirin

Label claim	Amount found	%Assay
Aspirin (150 mg)	8.2 mg	99.89

The results of the method development and validation show that the aspirin assay using Folin-Ciocalteu reagent is accurate, precise, and robust. The standard curve is linear over the range of concentrations tested, and the accuracy is within acceptable limits that is determined by %Recovery. The precision of the method is also within acceptable limits as determined by the %RSD that is less than 2. The LOD and LOQ values indicate that the method was sensitive.

CONCLUSION

Based on the experimental results, it can be concluded that the newly proposed spectrophotometric method for the determination of aspirin is rapid, accurate, economical. Because of its simplicity, sensitivity and selectivity the method viable alternative to the HPLC methods. As a result, the proposed method can be used for quality control purposes in the pharmaceutical industry.

ACKNOWLEDGEMENT

We are grateful to CMR college of pharmacy management for providing us with facilities such as equipment and chemicals to complete the work.

REFERENCES

1. Sutcliffe P, Connock M, Gurung T, et al. Aspirin for prophylactic use in the primary prevention of cardiovascular disease and cancer: a systematic review and overview of reviews. Southampton, UK: National Institute on Handicapped ResearchJournals Library; 2013Sep(Health Technology Assessment, No. 17.43.) Chapter 1, Introduction and background.
2. Awtry EH, Loscalzo J. Aspirin. Circulation.2000Mar14;101(10):1206-18. doi: 10.1161/01.cir.101.10.1206, PMID 10715270.
3. Khadak R, Birle R, Kayande N, Kushwah P. The Quantitative determination of the Aspirin content of tablets using UV and Visible wavelength Spectroscopy.
4. Suresh Kumar S, Jamadar LD, Bhat K, Musmade PB, Vasantharaju SG, Udupa N. Analytical method development and validation for aspirin. IntJ ChemTech Res. 2010;2(1):389-99.
5. Arif H, Aggarwal S. Salicyclic acid (aspirin)In: Statpearls; jan11.Treasure island, (FL):StatPearls publishing; 2022.
6. Dolores M, Hiplito EAM, Rosas JAG, Villaseor A, Arellano RL. Development and validation of an alternate stability-indicating UV spectrophotometric analytical method for aspirin in tablets.Indian J PharmSci. 2017Jan30;78(6):810-7. doi: 10.4172/pharmaceutical-sciences.1000187.
7. Murtaza G, Khan SA, Shabbir A, Mahmood A, Asad MH, Farzana Ket al.Development of a UV-spectrophotometric method for the simultaneous determination of aspirin and paracetamol in tablets. SciRes Essays.2011Jan18;6(2):417-21.
8. Chaudhari SS, Phalak SD. Development and validation of UVspectrophotometric method for simultaneous equation of Aspirin and omeprazole in tablet dosage form. PharmAnalActa.2019;11(1):618. doi: 10.35248/2153-2435.20.11.618.
9. Singh R, Khan T. Analytical method development and validation studies for the estimation of aspirin, clopidogrelbisulphate and rosuvastatin calcium in fixed dose combination (capsule) by UV spectroscopy. Indian Drugs.2017;54(6):43-7. doi: 10.53879/id.54.06.10885.
10. Fanes S, Rajput S. Method development and validation for simultaneousestimation of aspirin and lansoprazole in bulk and laboratorysample by differentUVspectrophotometrictechniques. IntJ PharmSci Res. 2015Aug1;6(8):3534.
11. Jones M, Thatcher RL. Spectrophotometric determination of aspirin, phenacetin, and caffeine in mixtures.AnalChem. 1951Jul1;23(7):957-60. doi: 10.1021/ac60055a008.
12. Gujarathi SC, Shah AR, Jagdale SC, Datar PA, Choudhari VP, Kuchekar BS. Spectrophotometric simultaneous determination of aspirin and ticlopidine in combined tablet dosage form by first order derivative spectroscopy, area under curve (AUC) and ratio derivative spectrophotometric methods.IntJ PharmSciRev Res. 2010Jul;3(1):115-9.
13. Game MD, Gabhane KB, Sakarkar DM. Quantitative analysis of clopidogrelbisulphate and aspirin by first derivative spectrophotometric method in tablets. Indian J PharmSci. 2010Nov;72(6):825-8. doi: 10.4103/0250-474X.84609, PMID 21969765.
14. Salehi A, Mohammady F, Kazemipour M, Ansari M. Simultaneous UV-vis spectrophotometric determination of aspirin and methocarbamol in pharmaceutical preparations. Res PharmSci. 2012Sep2;7(5):669.