Alqalam Journal of Medical and Applied Sciences. 2025;8(2):832-835
https://doi.org/10.54361/ajmas.258240

Original article

Comparative Quantitative Study of Acetyl Salicylic Acid in Aspirin Samples Using Spectrophotometry and Volumetric

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Abstract
This study focuses on the quantification of acetylsalicylic acid (ASA) in aspirin tablets using two analytical approaches: back-titration and spectrophotometric analysis. Significant variations in ASA concentrations were observed across different samples. The back-titration method recorded concentrations ranging from 33.68% to 75.27%, while spectrophotometric analysis yielded values between 26.27% and 74.65%. These discrepancies highlight potential factors such as formulation inconsistencies, manufacturing quality, or chemical interactions during analysis. A comparative evaluation of the two methods reveals that the spectrophotometric method demonstrated higher precision and ease of execution, making it suitable for routine pharmaceutical analysis. However, both methods showed limitations in accuracy. The findings suggest variability in ASA concentrations among different commercial brands, which remain largely within official standards but warrant further investigation to ensure consistency and efficacy. The study underscores the need for refining analytical methods to enhance reliability and calls for improved manufacturing protocols to maintain quality control in pharmaceutical products.
Keywords. Aspirin. Titration, Spectrophotometer, Salicylic Acid, Back-Titration.

Introduction

Drug analysis is an essential component of ensuring the quality of pharmaceuticals worldwide, helping to ensure the safety and efficacy of medications before they are used in patient treatment. Over the years, analytical techniques have developed to include a variety of methods that contribute to determining the active ingredients in drugs. High-performance liquid chromatography (HPLC) is one of the most precise and widely used techniques for drug analysis. However, the high cost and technical demands of HPLC can make it inaccessible in certain laboratories, leading to a need for alternative, cost-effective methods that provide accurate results [1]. Aspirin, or acetylsalicylic acid, is one of the most widely used medications globally for treating pain, fever, and inflammation, in addition to its preventive role in reducing the risk of cardiovascular diseases [2]. For ensuring its therapeutic efficacy, it is crucial to determine the concentration of its active ingredient, salicylic acid [3]. Therefore, determining the purity of aspirin using precise techniques has become necessary to ensure its quality in the market. This study involves the collection of aspirin samples from pharmacies in Tripoli, Libya, to assess the quality of aspirin and determine the concentration of its active ingredient (salicylic acid) in these samples. Volumetric methods (direct and back titration) and spectrophotometric methods were employed as alternatives for aspirin analysis, due to their lower cost and simpler implementation compared to more complex analytical methods [4]. The volumetric method, whether direct or back titration, is one of the oldest and most widely used methods for drug analysis, where a standard solution is added gradually to the unknown solution until the equivalence point is reached, allowing for the calculation of the active ingredient concentration [5]. Meanwhile, spectrophotometric methods rely on measuring the absorption of light at specific wavelengths, providing a fast and accurate way to determine the concentration of salicylic acid in aspirin. This method is straightforward to apply and less expensive compared to others, as it does not require complex setups or costly equipment [6].
The primary aim of this study is to use volumetric methods and spectrophotometric techniques to determine the concentration of salicylic acid in aspirin samples collected from local pharmacies in Tripoli. The study will evaluate the accuracy and reliability of these methods in estimating the active ingredients in aspirin, providing a cost-effective and efficient alternative for drug quality control. This study contributes to offering practical solutions for laboratories facing economic challenges in using complex techniques, striving to enhance drug quality control and ensure the safety and efficacy of pharmaceuticals in the market.

Methods

Equipment

Various equipment was used in the experiment, including conical flasks, volumetric flasks, burettes, pipettes, glass stirring rods, and flasks. A sensitive balance was employed to accurately measure the masses, in addition to a spectrophotometer used for measuring light absorbance.

Materials

The materials used in the preparation and reactions included phenolphthalein, ethanol, sodium hydroxide (0.1M), salicylic acid, ferric chloride (0.02M), and aspirin tablets.

Received*: 01-03-2025 -* ***Accepted****: 29-04-2025 -* ***Published****: 07-05-2025*

Sample collection and preparation

Fourteen samples of aspirin were collected from various pharmaceutical companies, both European and Arab, from pharmacies in the city of Tripoli, including those located in Souq al-Jummah, 11 June Street, and Al-Nasr Street.

Preparation of Standard Solutions

The standard solutions were prepared as follows:

- Preparation of Phenolphthalein Indicator: 0.5 grams of phenolphthalein were dissolved in 50 mL of ethanol (95%), and the volume was made up to 100 mL with distilled water.
- Preparation of Sodium Hydroxide Solution: A 0.1M sodium hydroxide solution was prepared by dissolving 4 grams of sodium hydroxide in a 100 mL volumetric flask.
- Preparation of Salicylic Acid Solution: A 0.1M salicylic acid solution was prepared by dissolving 1.38 grams of salicylic acid in a 100 mL volumetric flask. A series of diluted standard solutions was then prepared with concentrations ranging from 0.001M to 0.006 M.
- Preparation of Ferric Chloride Solution: A 0.02M ferric chloride solution was prepared by dissolving 1.6 grams of ferric chloride in a 500 mL volumetric flask.

Titration process for the sample

To determine the concentration of acetylsalicylic acid in the aspirin samples, an aspirin tablet was weighed, then finely ground and dissolved in 10 mL of ethanol. After that, 25 mL of distilled water and 3 drops of phenolphthalein indicator were added. The solution was titrated with sodium hydroxide solution until a pink color appeared, and this process was repeated three times for each sample.

The titration process involves the oxidation of salicylic acid, where the solution is initially acidic. With the gradual addition of the base, the pH increases until it reaches the endpoint, where the number of millimoles of the base equals the number of millimoles of the acid. At this point, the color changes from colorless to pink, and the pH of the solution reaches. The following equation was used to calculate the concentration of salicylic acid:

$$M_{NaOH} \times V_{NaOH} = M_{SA} \times V_{SA}$$

The concentration of the acid was then converted from mol/L to mg/L (PPM) using the formula:

$$ppm = \frac{molarity}{M.wt \times 1000}$$

To calculate the purity, the following equation was used:

$$purity\% = \frac{(mass\ of\ acid)}{(mass\ of\ aspirin)} \times 100$$

The reaction equation between salicylic acid and sodium hydroxide was as follows:

$C_7H_6O_3+NaOH \rightarrow C_6H_4(OH)CO_2Na+H_2O$

Spectrophotometric analysis of the sample

This technique relies on converting acetylsalicylic acid into salicylic acid by adding sodium hydroxide. After breaking the bond, salicylic acid and acetic acid are formed. A complex is then formed with iron salt, which appears purple. This complex is subjected to ultraviolet light in the range of 200 to 800 nanometers to determine the maximum wavelength.

Procedure

The sample measurement process using the spectrophotometer was divided into two parts. In the first part, a series of concentrations of the original salicylic acid solution (0.1M) were prepared in 100 mL volumetric flasks, with concentrations ranging from 0.001M to 0.006 M. Then, 20 mL of ferric chloride solution was added to each flask, and the volume was made up to the calibration mark with distilled water. The absorbance of each prepared solution was measured. Using the straight-line equation derived from these measurements, the concentration of acetylsalicylic acid in the samples was determined. In the second part, the sample was dissolved in 5 mL of sodium hydroxide in a 100 mL volumetric flask, and the volume was completed with distilled water. Then, 49 mL of ferric chloride solution (0.02M) was added, resulting in a purple-colored solution. The absorbance of this solution was measured, and the previous equation was used to calculate the actual concentration of the active ingredient in the samples. This process was repeated for each of the studied samples (Chiriac, A., & Rusu).

Received*: 01-03-2025 -* ***Accepted****: 29-04-2025 -* ***Published****: 07-05-2025*

Calibration Curve

A calibration curve was constructed to determine the concentration of acetylsalicylic acid (ASA) in the aspirin samples. Standard solutions of salicylic acid with known concentrations ranging from 0.001M to 0.006M were prepared and reacted with ferric chloride. The absorbance of each solution was measured at the maximum wavelength of 530 nm, and the results were used to plot the curve. The curve exhibited a strong linear relationship between absorbance and concentration, as indicated by the equation 0.1593x + 1.5057y and a correlation coefficient (R^2 = 0.9676). This curve served as the basis for determining ASA concentrations in the test samples by comparing their absorbance values to the calibration line.

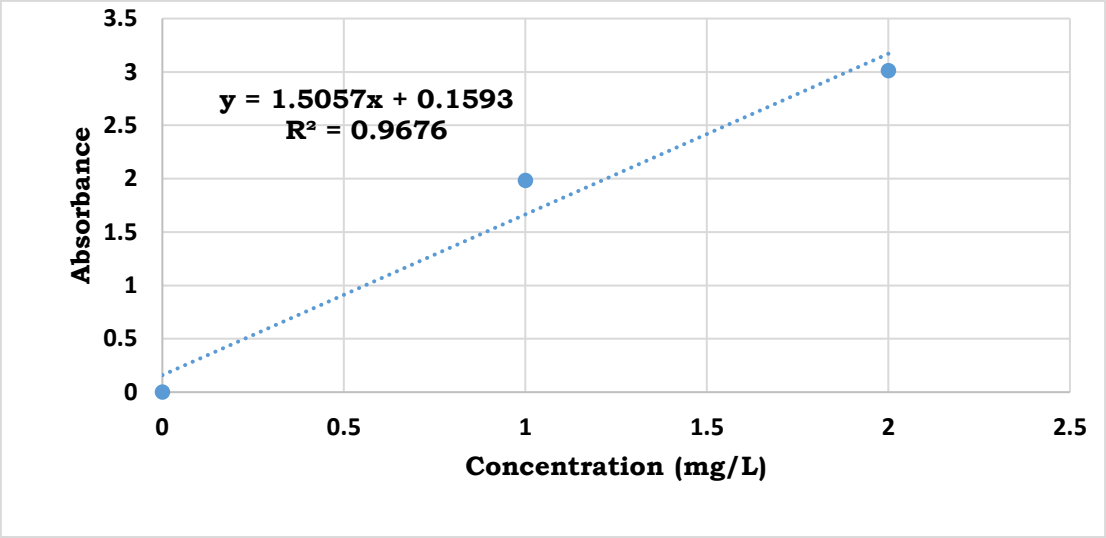

Figure 1. Spectrophotometric calibration curve.

Results and discussion

A total of 14 samples of aspirin tablets were analyzed to determine the concentration of acetylsalicylic acid using two methods: Back-Titration and Spectrophotometric Method. The results are presented in Table 1.

Table 1. The results of the Back-titration and Spectrophotometric method

Number	Weight g/tablet	% Back titration	% Spect.
1	0.204	66.17	55.07
2	0.182	75.27	74.65
3	0.132	73.48	63.66
4	0.148	52.70	42.15
5	0.174	67.24	38.64
6	0.160	41.87	49.06
7	0.134	47.01	66.55
8	0.138	57.14	55.89
9	0.115	44.66	63.14
10	0.133	41.35	53.05
11	0.156	40.38	54.78
12	0.317	47.63	68.84
13	0.110	66.36	55.62
14	0.187	33.68	26.27

The findings reveal a notable variation in the concentration of acetylsalicylic acid (ASA) across the analyzed samples. For example, sample 2 exhibited the highest percentage of back titration at 75.27%, whereas sample 14 showed the lowest percentage at 33.68%. These discrepancies may be attributed to differences in manufacturing quality, formulation composition, or variability in active ingredient distribution within the tablets [7,8]. Similarly, results obtained from the spectrophotometric method indicate that the highest recorded concentration was 74.65% (sample 2), while the lowest was 26.27% (sample 14). These variations suggest potential factors such as the presence of impurities, inconsistencies in tablet dissolution, or chemical interactions that could impact concentration estimations [9].

When compared with previous studies, such as Smith et al., which reported an average ASA concentration of 78% in aspirin tablets, it is evident that several samples in this study yielded lower values [7]. This deviation highlights the need for a comprehensive assessment of manufacturing protocols and quality

Received*: 01-03-2025 -* ***Accepted****: 29-04-2025 -* ***Published****: 07-05-2025*

control measures to ensure consistency in drug formulation [10,11]. Additionally, results confirm that back titration demonstrates lower accuracy relative to the Spectrophotometric method, which has been widely recognized for its precision in pharmaceutical analysis [12]. Prior research by Jones & Lee established that spectrophotometric analysis produced results comparable to back titration but with improved accuracy, further supporting the reliability of this method for ASA quantification [13].

Conclusion

This study underscores the variability in ASA concentrations across different aspirin samples, which may have implications for the effectiveness of the medication. To ensure consistency in pharmaceutical formulations, future research should investigate the factors contributing to these discrepancies, refine analytical methodologies to enhance precision, and optimize manufacturing and storage conditions to maintain product quality.

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الملخص

تركز هذه الدراسة على تحديد كمية حمض أسيتيل الساليسيليك في أقراص الأسبرين باستخدام نهجين تحليليين: المعايرة العكسية والتحليل الطيفي الضوئي. لوحظت اختلافات كبيرة في تركيزات حمض أسيتيل الساليسيليك عبر عينات مختلفة. سجلت طريقة المعايرة العكسية تركيزات تتراوح بين 33.68٪ و 75.27٪، بينما أسفر التحليل الطيفي الضوئي عن قيم تتراوح بين 26.27٪ و 74.65٪. تسلط هذه التناقضات الضوء على عوامل محتملة مثل عدم اتساق التركيبة أو جودة التصنيع أو التفاعلات الكيميائية أثناء التحليل. يكشف التقييم المقارن للطريقتين أن الطريقة الطيفية الضوئية أظهرت دقة أعلى وسهولة في التنفيذ، مما يجعلها مناسبة للتحليل الصيدلاني الروتيني. ومع ذلك، أظهرت كلتا الطريقتين قيودًا في الدقة. تشير النتائج إلى وجود تباين في تركيزات حمض أسيتيل الساليسيليك بين العلامات التجارية المختلفة، والتي لا تزال إلى حد كبير ضمن المعايير الرسمية ولكنها تستحق المزيد من البحث لضمان الاتساق والفعالية. وتؤكد الدراسة على الحاجة إلى تحسين الأساليب التحليلية لتعزيز الموثوقية وتدعو إلى تحسين بروتوكولات التصنيع للحفاظ على مراقبة الجودة في المنتجات الصيدلانية.

Received*: 01-03-2025 -* ***Accepted****: 29-04-2025 -* ***Published****: 07-05-2025*