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THE SYNTHESIS AND CHARECTERIZATION OF ACETYLSALICYLIC ACID (ASPIRIN)

Pratik Pradip Narwade*1, Vishal Gajanan Hule*2

*1UG Student, Department Of Pharmacy, Karmayogi Tatyasaheb Bondre Institute Of Pharmacy, Chikhli, Maharashtra, India.

*2Assistant Professor, Department Of Pharmacy, Karmayogi Tatyasaheb Bondre Institute Of Pharmacy, Chikhli, Maharashtra, India.

ABSTRACT

This research deals with the synthesis of acetylsalicylic acid (aspirin) through the esterification reaction of salicylic acid with acetic anhydride in the presence of a catalyst, sulfuric acid. The product was purified by recrystallization and identified through the determination of its melting point and the use of thin layer chromatography. The results confirmed the successful synthesis of aspirin with good purity and yield

Keywords: Aspirin, Acetylsalicylic Acid, Esterification, Recrystallization, Thin Layer Chromatography.

I. INTRODUCTION

Aspirin (Acetylsalicylic Acid) is a common analgesic, antipyretic, and anti-inflammatory agent. It is prepared through esterification between salicylic acid and acetic anhydride. This Experiment illustrates a basic organic synthesis and features elementary techniques practiced in pharmaceutical chemistry for compound analysis and purification

OBJECTIVES –

- To synthesize acetylsalicylic acid
- To purify the product by recrystallization
- To characterize the compound using melting point analysis, TLC.

Aspirin, or acetylsalicylic acid, is one of the maximum used drugs in the world due to its painkillers, antipyretic and anti -inflammatory properties. The first at the end of the nineteenth century synthesized, it remains a foundation stone in both scientific and to pure treatment. The synthesis of aspirin is a classic natural chemistry test that shows large standards with respect, processing and compound properties.The reaction forces acetizing salicylic acid with vinegar anhydride in the presence of a acid catalyst, usually sulphur or phosphoric acid. This technique has an effect in the formation of aspirin and acetic acid as a by -product. After synthesis, the product is cleaned, usually through recrystallization, followed by the use of techniques that include melting factor analysis and infrared (IR) spectroscopy that confirms its identification and checks its purity.

This research should synthesize aspirin in the laboratory and evaluate the accuracy of physical and analytical strategies, which strengthen the natural reaction system and analytical chemistry information.

II. METHODOLOGY

CHEMICALS –

- SALICYLIC ACID
- ACETIC ANHYDRIDE
- CON. SULPHURIC ACID
- DISTIL WATER
- ETHANOL

PROCEDURE -

1. Weighed 2 g of salicylic acid and placed it in dry conical flask
2. Added 4 ml of acetic anhydride
3. Added 5 drops of con. Sulphuric acid as catalyst
4. Heated the mixture in water bath at 70*C for 15 minutes
5. Removed the flask and allowed it cool to room temperature

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6. Poured the reaction into 40 ml of cool distilled water.
7. Placed the mixture in an ice bath to complete precipitation.
8. Filtered the solid product using vacuum filtration

III. ANALYSIS

The synthesis of aspirin becomes based at the esterification response among salicylic acid and acetic anhydride, catalysed by an acid. Upon of entirety, the product turned into purified thru recrystallization, and several strategies had been used to investigate its purity and affirm its identity.

1. Yield Analysis

The mass of the final product was measured and in comparison, to the theoretical yield primarily based at the preliminary quantity of salicylic acid. The percent yield affords insight into the performance of the reaction. A lower-than-anticipated yield may be attributed to incomplete reaction, product loss at some point of transfer or filtration, or solubility losses at some point of recrystallization.

Balanced Reaction-

$C_7 H_6 O_3$ (salicylic acid) + $C_4 H_6 O_3$ (Acetic Anhydride) → $C_9H_8O_4$(Aspirin) + CH3COOH

Theoretical yield- 2.61

Practical yield- 1.90

Percentage yield- practical yield/theoretical yield*100

1.90/2.61*100

72.8%

Percentage Yield = 72.8%

Table 1:

Interpreting Result	Percentage
Good yeild	70-90
Low yield	<70
High yield	>100

2. Melting Point Determination

Purity of synthesized aspirin was evaluated by measuring the melting point. Pure aspirin has a melting limit of approximately 134-135° C. Possible deviations or extension in the melting limit indicates the presence of impurities. If the observed melting point is quite low or it has a wide area, it indicates that the product may still contain unattainable salicylic acids or other impurities.

The Observed Melting Point - 136°

3. Thin Layer Chromatography

Material Used-

1. TLC Plate – Silica Gel
2. Solvent- Acetone
3. Samples- Standard Salicylic acid solution, Aspirin solution, crude product
4. Visualizing Agent- UV Lamp

Procedure

1. place all three samples on the same baseline on a TLC plate.
2. Create a plate in a TLC room using a solvent system.
3. Take away the plate when it is about 1 cm from the solvent's edge.
4. Envision UV light or apply $FeCl_3$ spray:

a. Salicylic acid imparts a purple hue with facl because of the phenol group.
b. Aspirin does not interact with $Facl_3$ (phenol undergoes acetylation)

$$Rf = \frac{\text{Distance travel by Compound(mm)}}{\text{Distance travel by solvent front(mm)}}$$

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Table 2:

Samples	Rf value	observation
Salicylic acid	0.30	Dark Spot react with $fecl_3$
Standard Aspirin	0.48	No reaction
Synthesized product	0.41	No reaction

IV. RESULT

Table 3:

TEST	LITERATURE VALUE	OBSERVED
Physical Appearance	White crystals	White crystals
Yeild Analysis	75-80%	72%
Melting Point	134-135°	136°
TLC	0.45-0.50	0.41

V. CONCLUSION

The synthesis of aspirin (acetylsalicylic acid) turned into efficaciously done through the esterification reaction among salicylic acid and acetic anhydride within the presence of an acid catalyst. The response yielded a white crystalline product, confirming the formation of aspirin. The purity and identification of the synthesized compound had been confirmed thru melting factor dedication and spectroscopic techniques consisting of IR spectroscopy, which confirmed function peaks corresponding to the ester and carboxylic acid practical groups.

Overall, the test tested the principles of organic synthesis, along with response conditions, purification techniques, and compound characterization. Minor deviations in yield or melting factor can be attributed to incomplete reaction or impurities, however a success isolation and analysis of aspirin affirmed the effectiveness of the synthesis approach

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