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TO SYNTHESIS ASPIRIN FROM SALICYLIC ACID AND EVALUATE ITS PURITY

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ABSTRACT

The present project focuses on the synthesis of Aspirin (acetylsalicylic acid) from Salicylic acid and the evaluation of its purity using standard pharmaceutical methods. Aspirin is a widely used non-steroidal antiinflammatory drug (NSAID) known for its analgesic, antipyretic, antiinflammatory, and antiplatelet properties. Due to its extensive therapeutic applications, the study of its preparation and quality assessment is of great importance in pharmaceutical sciences.

The synthesis is carried out by an acetylation reaction in which salicylic acid reacts with acetic anhydride in the presence of an acid catalyst such as sulfuric acid or phosphoric acid. During this reaction, the phenolic hydroxyl group of salicylic acid is converted into an ester group, forming aspirin, while acetic acid is produced as a byproduct. The reaction is performed under controlled temperature conditions using a water bath to ensure proper completion.

The crude product obtained is purified by recrystallization using ethanol to remove impurities and obtain pure aspirin crystals. The purity of the synthesized aspirin is evaluated using melting point determination, Ferric chloride test, and Thin Layer Chromatography (TLC). These analytical techniques help in confirming the identity and quality of the product. This project provides a clear understanding of drug synthesis, purification techniques, and quality control methods used in pharmaceutical chemistry

KEYWORDS: *Aspirin, Salicylic acid, Acetylation, Recrystallization, Purity evaluation, TLC, Melting point*

1. INTRODUCTION

Acetylsalicylic acid, commonly known as Aspirin, reduces pain, fever, and inflammation while also lowering the risk of heart attacks and strokes. It is among the most widely used medications. Its effectiveness arises from its analgesic, antipyretic, and anti-inflammatory properties. In the small intestine, acetylsalicylic acid breaks down into salicylic acid, which is then absorbed into the bloodstream. Salicylic acid can irritate.

the lining of the mouth, esophagus, and stomach, potentially leading to hemorrhaging due to its phenolic and carboxylic acid groups. The Bayer company discovered that the ester of salicylic acid (acetylsalicylic acid) is less irritating than the original salicylic acid. However, it still presents side effects, such as stomach lining irritation and hemorrhaging; to reduce these effects, commercial Aspirin often includes coatings and buffering agents.

SYNTHESIS OF ASPIRIN (Acetylsalicylic Acid)

Aspirin is produced by combining salicylic acid with excess acetic anhydride (the oacetylation of salicylic acid) and a small amount of a strong acid catalyst, which accelerates the reaction. A strong acid catalyst, such as 85% phosphoric acid or concentrated sulfuric acid, promotes the esterification reaction, resulting in the formation of white crystals. Water is then added to remove the surplus acetic anhydride. In summary, the reaction involves a carboxylic acid and an acid anhydride, which together form an ester.

- **Reaction Equation**

Salicylic acid+Acetic anhydride→H_3PO_4Aspirin+Acetic acid

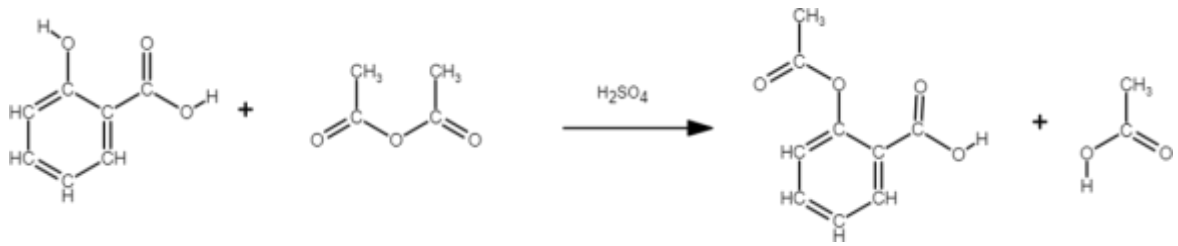

Figure : Synthesis of Aspirin.

Aspirin has very low solubility in cold water and is utilized for precipitation. Any unreacted salicylic acid is considered an impurity due to its similarly low solubility in cold water.
Dissolved unreacted substances, such as acetic anhydride and acetic acid, remain in the water. Vacuum filtration effectively separates the crystalline aspirin from the reaction mixture, excluding unreacted salicylic acid. The synthesized aspirin should be evaluated for the presence of contaminating salicylic acid. The purity of crude and recrystallized aspirin can be determined using iron (III) chloride ($FeCl_3$).

This reaction is an example of esterification or acetylation reaction in organic chemistry. In this reaction, hydroxyl group present in salicylic acid gets acetylated by acetic anhydride resulting in formation of acetyl salicylic acid, commonly known as aspirin. Concentrated sulphuric acid acts as catalyst and increases the rate of reaction.

The synthesized aspirin is obtained in the form of white crystalline powder after crystallization and purification process. Crystallization is an important step because it helps in separation and purification of aspirin from impurities and unreacted chemicals. The obtained crystals are filtered, washed, dried, and further evaluated for purity.

Aspirin possesses important physical and chemical properties. It appears as white crystalline powder with slight vinegar-like odour due to formation of acetic acid. Aspirin is weak acidic compound because it contains carboxylic acid functional group. It is slightly soluble in water but freely soluble in ethanol and alkaline solutions. The molecular formula of aspirin is $C_9H_8O_4$ and molecular weight is 180.16 g/mol.

The melting point of aspirin is near135□C
Melting point determination is one of the most important methods used for evaluation of purity of synthesized aspirin. Pure aspirin shows melting point near the standard value, whereas impurities lower and broaden the melting point range.

Aspirin possesses several medicinal and therapeutic applications. It acts as analgesic drug and helps in relief of headache, toothache, muscle pain, and body pain. Aspirin also acts as antipyretic agent and reduces fever by acting on temperature regulating centre of brain. Because of its anti-inflammatory property, aspirin is widely used in treatment of arthritis, rheumatic disorders, and inflammatory conditions.

Another important property of aspirin is antiplatelet activity. Low doses of aspirin prevent blood clot formation by inhibiting platelet aggregation. Therefore, aspirin is commonly used in prevention of heart attack, stroke, and cardiovascular diseases.

Salicylic acid, which is used as starting material in aspirin synthesis, also possesses pharmaceutical importance. It is widely used in treatment of acne, dandruff, psoriasis, warts, and various skin disorders because of its keratolytic and antibacterial properties.

The synthesis of aspirin is one of the most commonly performed practical experiments in pharmaceutical chemistry and organic chemistry laboratories. This experiment helps students understand esterification reaction, acetylation process, crystallization, filtration, recrystallization, drying techniques, and purity evaluation methods. It also provides practical knowledge about handling of chemicals, use of catalysts, heating process, and laboratory safety precautions.

Purity of synthesized aspirin can be evaluated by various methods such as physical appearance, melting point determination, ferric chloride test, and percentage yield calculation. Ferric chloride test acts as confirmatory test for aspirin synthesis. After hydrolysis, aspirin gives violet coloration with ferric chloride solution due to formation of salicylic acid.

Percentage yield is another important parameter used to determine efficiency of synthesis process. High percentage yield indicates successful reaction and proper experimental conditions.

Thus, synthesis of aspirin from salicylic acid is an important pharmaceutical practical experiment that demonstrates preparation, purification, characterization, and evaluation of one of the most widely used drugs in medicinal and pharmaceutical science.

DRUG PROFILE

Drug Profile: Aspirin (Acetylsalicylic Acid)

Chemical Identity

- Molecular formula: $C_9H_8O_4$; white crystalline powder. • It is an ester derivative of salicylic acid, formed by acetylation.

Mechanism of Action

1. Irreversibly inhibits cyclooxygenase (COX-1 and COX-2) enzymes.
2. Reduces prostaglandin and thromboxane synthesis, lowering pain, fever, inflammation, and platelet aggregation.

Therapeutic Uses

1. Acts as an analgesic, antipyretic, and anti-inflammatory agent.
2. Low-dose aspirin is widely used for cardiovascular protection by preventing clot formation.

Safety and Limitations

a. Common side effects include gastric irritation and risk of bleeding.
b. Contraindicated in children with viral infections (risk of Reye's syndrome) and patients with peptic ulcers or bleeding disorders

2. LITERATURE

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5. International Journal of Pharmaceutics (2020) – Stability and degradation of acetylsalicylic acid.
6. British Journal of Pharmacology (2016) – Mechanism of action of aspirin as COX inhibitor.
7. Felix Hoffmann,et;al (1897) :- Aspirin was first synthesized from salicylic acid by acetylation reaction using acetic anhydride. The synthesized aspirin showed analgesic and antipyretic properties and became one of the most widely used pharmaceutical drugs.
8. Vane J.R,et;al (1971) :- This study reported that aspirin inhibits cyclooxygenase (COX) enzyme and reduces prostaglandin synthesis responsible for pain, fever, and inflammation. The study explained the mechanism of action of aspirin.
9. Rainsford K.D,et;al (2004) :- The review highlighted the pharmaceutical and medicinal importance of aspirin. It reported analgesic, antipyretic, anti-inflammatory, and antiplatelet properties of aspirin.

3. AIM & OBJECTIVES

AIM :- To synthesize aspirin from Salicylic acid and evaluate Its purity.

OBJECTIVES

1. To understand the synthesis of Aspirin in laboratory conditions.
2. To study the reaction of Salicylic Acid with Acetic Anhydride.
3. To learn the role of Sulfuric Acid as a catalyst in acetylation reaction.
4. To perform the synthesis of aspirin using proper laboratory techniques.
5. To purify the crude product by recrystallization method using ethanol

4. PLAN OF WORK

1. Study of synthesis of Aspirin from Salicylic acid.
2. Collection of literature & understanding reaction principle (acetylation).
3. Arrangement of chemicals and laboratory apparatus.
4. Preparation of reaction mixture (S"licy'Ic acid + Acetic anhydride + catalyst).
5. Heating in water bath under controlled temperature.
6. Cooling to obtain aspirin crystals.
7. Filtration and recrystallization for purification.
8. Testing of purity (Melting point, $FeCl_3$'test, TLC).
9. Calculation of percentage yield.
10. Observation recording and final results writing.

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5. MATERIAL & METHODS

5.1 Materials

SR.NO	Chemicals	Quantity	Purpose
1	Salicylic acid	2 g	Reactant
2	Acetic anhydride	4 mL	Acetylating agent
3	Concentrated sulfuric acid	4–5 drops	Catalyst
4	Distilled water	q.s.	Washing and recrystallization
5	Ethanol	10 mL	Recrystallization
6	Ferric chloride solution	Few drops	Purity test

5.2 Instruments

SR.NO	Instrument	Use
1	Beaker	Reaction vessel
2	Measuring cylinder	Measurement of liquids
3	Glass rod	Stirring
4	Water bath	Heating
5	Funnel	Filtration
6	Filter paper	Separation of crystals
7	Weighing balance	Accurate weighing
8	Melting point apparatus	Determination of melting point

6. FORMULATION TABLE

Formula for Synthesis of Aspirin

Ingredients	Quantity Used	Role
Salicylic Acid	2 g	Starting material
Acetic Anhydride	4 mL	Acetylating agent
Concentrated Sulfuric Acid	4–5 drops	Catalyst
Distilled Water	q.s.	Washing and precipitation
Ethanol	10 mL	Recrystallization solvent

7. METHOD OF PREPARATION

Step 1: Preparation of Reaction Mixture

2 g of salicylic acid was accurately weighed and transferred into a dry conical flask. To this, 4 mL of acetic anhydride was added carefully.

Step 2: Addition of Catalyst

4–5 drops of concentrated sulfuric acid were added slowly into the reaction mixture while stirring continuously.

Step 3: Heating

The reaction mixture was heated on a water bath at approximately 60–70°C for 15–20 minutes with occasional stirring.

Step 4: Formation of Aspirin Crystals

After completion of heating, the reaction mixture was cooled and 20 mL of cold distilled water was added slowly. The flask was kept in an ice bath for crystallization.

Step 5: Filtration

The formed crystals were collected by filtration using filter paper and washed with cold distilled water to remove impurities.

Step 6: Recrystallization

The crude aspirin crystals were dissolved in minimum quantity of warm ethanol and recrystallized by cooling to obtain pure aspirin crystals.

Step 7: Drying

The purified crystals were dried between filter papers and stored in an airtight contain

8.EVALUATION PARAMETERS

8.1 Physical Appearance

The synthesized aspirin was observed visually for color, texture, and crystal nature. **Observation** White crystalline powder was obtained.

8.2 Determination of Percentage Yield Formula

Percentage Yield = (Practical Yield / Theoretical Yield) × 100

Calculation

Parameter	Value
Weight of salicylic acid	2 g
Theoretical yield of aspirin	2.61 g
Practical yield obtained	2.20 g

Percentage Yield

Percentage Yield = (2.20 / 2.61) × 100 Percentage Yield = 84.29%

8.3 Melting Point Determination

The melting point of synthesized aspirin was determined using a melting point apparatus.

Sample	Melting Point
Standard aspirin	135°C – 136°C
Synthesized aspirin	134°C – 136°C

Interpretation

The obtained melting point was close to the standard value, indicating good purity of aspirin.

8.4Ferric Chloride Test Procedure

A small quantity of synthesized aspirin was dissolved in distilled water and few drops of ferric chloride solution were added. **Observation** Very light violet coloration was observed.

Interpretation

The absence or very light violet color indicates minimal presence of unreacted salicylic acid and confirms good purity.

8.5 Solubility Test

The synthesized aspirin was tested for solubility in water and ethanol. Solvent Observation
Water Slightly soluble Ethanol Soluble

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RESULT

The aspirin synthesized from salicylic acid was obtained as white crystalline powder. The prepared aspirin showed satisfactory percentage yield and melting point close to the standard value. The ferric chloride test indicated very low amount of unreacted salicylic acid, confirming good purity of the synthesized product.

RESULT TABLE

Evaluation Parameter Observation/Result

Appearance	White crystalline powder
Percentage Yield	84.29%
Melting Point	134°C – 136°C
Ferric Chloride Test	Very light violet color
Solubility in Water	Slightly soluble Solubility in Ethanol Soluble

DISCUSSION

The synthesis of aspirin was successfully carried out by acetylation of salicylic acid using acetic anhydride in the presence of concentrated sulfuric acid catalyst. The reaction proceeded effectively under controlled heating conditions.

The crude aspirin obtained after crystallization contained minor impurities which were removed by recrystallization using ethanol. The purified aspirin appeared as white crystalline powder.

SUMMARY

The present study involved the synthesis of aspirin from salicylic acid using acetic anhydride and sulfuric acid catalyst. Aspirin was prepared by acetylation reaction followed by purification through recrystallization.

The synthesized aspirin was evaluated for physical appearance, percentage yield, melting point, ferric chloride test, and solubility. The product obtained was white crystalline powder with satisfactory purity and yield.
The melting point and ferric chloride test confirmed successful synthesis and purification of aspirin.

CONCLUSION

The study successfully demonstrated the synthesis of aspirin from salicylic acid by acetylation reaction. The prepared aspirin was purified effectively by recrystallization method.

Evaluation parameters such as melting point, ferric chloride test, and percentage yield confirmed that the synthesized aspirin possessed good purity and quality. Therefore, aspirin can be synthesized effectively in laboratory conditions using simple and economical methods.

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