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MICROWAVE ASSISTED SYNTHESIS OF BENZOIC ACID

Aditya A. Mali, * Srinivas R. Mane, Sanjay K. Bais, S. M. Kazi
Fabtech College of Pharmacy, Sangola
Tal-Sangola, Dist.-Solapur
Maharashtra -413307

ABSTRACT

Microwave-assisted synthesis presents an expedient approach for the conversion of benzanilide into benzoic acid, a key organic compound with diverse applications. In this study, we investigate the microwave-assisted transformation of benzanilide to benzoic acid using concentrated sulfuric acid as the catalyst. The reaction was conducted under controlled microwave irradiation conditions, optimizing parameters such as temperature, reaction time, and reagent concentration to maximize yield and purity. The efficiency and selectivity of the microwave-assisted synthesis route highlight its potential for scalable production of benzoic acid with reduced reaction times and environmental impact compared to conventional methods. This methodology offers a promising avenue for the sustainable synthesis of benzoic acid, facilitating its utilization in pharmaceutical, agrochemical, and industrial sectors.

Microwave heating provided precise control over the reaction temperature and ensured uniform heating throughout the reaction mixture, resulting in accelerated reaction rates and decreased response times in comparison to traditional heating techniques. Furthermore, the scalability of the microwave-assisted synthesis was evaluated, indicating its potential for large-scale production. The environmentally friendly nature of microwave heating, coupled with the efficiency of the catalytic system, makes this method an attractive alternative to conventional synthesis routes for benzoic acid. Overall, this study highlights the feasibility and usefulness of microwave-aided synthesis for the production of benzoic acid, with implications for both academic research and industrial applications.

Keywords: *Microwave irradiation, sulfuric acid, microwave oven and benzoic acid.*

*Corresponding Author Email: - adityamali2907@gmail.com

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INTRODUCTION

The field of organic synthesis has undergone a transformative evolution in recent years, driven by the incessant pursuit of more sustainable, efficient, and expedited synthetic methodologies. Among these advancements, microwave-assisted synthesis has emerged as a revolutionary technique, reshaping the landscape of chemical design and fabrication. This innovative approach leverages microwave irradiation as a heating source in chemical reactions, showcasing unprecedented advantages such as significantly shortened reaction times, enhanced yields, and the exploration of novel chemical pathways. Microwave-assisted synthesis offers a paradigm shift from traditional heating methods, promising a more environmentally friendly and time-efficient alternative. As the demand for greener and more sustainable synthetic routes intensifies, this technique has garnered attention for its potential to address these challenges effectively. In this comprehensive review, we aim to delve into the fundamental principles that underlie microwave-assisted organic synthesis, elucidating the mechanisms that govern its efficacy. Furthermore, we will explore the diverse range of organic reactions that have benefited from microwave irradiation, spanning from classic transformations to cutting-edge methodologies. [1]

By shedding light on the impact of microwave-assisted synthesis on reaction selectivity, stereoselectivity, and overall process optimization, this review attempts to offer a thorough understanding of its applications in the realm of organic chemistry. The focus of our study lies in the microwave-assisted conversion of benzanilide to benzoic acid, a reaction that exemplifies the potential of this technique in the sustainable production of valuable organic compounds. [2] Through an exploration of the reaction optimization, mechanistic insights, and characterization of the resulting benzoic acid, we seek to contribute valuable knowledge to the broader field of microwave-assisted synthesis of organic matter. As we embark on this journey through the intricacies of microwave-assisted synthesis, we aim to highlight the significance of this methodology in addressing the current challenges faced by the scientific community. By providing a comprehensive overview of its principles, mechanisms, and diverse applications, this review aims to further the continued advancement and adoption of microwave-assisted synthesis as a green and efficient tool in organic chemistry. [3]

Microwave-assisted synthesis is an innovative and efficient method widely used in organic chemistry to accelerate chemical reactions. One notable application of this technique is the synthesis of benzoic acid from benzanilide. Benzoic acid, a simple aromatic carboxylic acid, has various applications in food preservation, pharmaceuticals, and cosmetics.[4]

The traditional synthesis of benzoic acid from benzanilide involves prolonged heating under reflux conditions with strong acids, which is time-consuming and energy-intensive. Microwave-assisted synthesis offers a greener alternative by significantly reducing reaction times and enhancing yields through the use of microwave irradiation. This method utilizes the ability of microwaves to rapidly heat polar solvents and reagents, thereby accelerating reaction rates.[5]

In the context of synthesizing benzoic acid from benzanilide, sulfuric acid is often employed as a catalyst to facilitate the hydrolysis of the amide group in benzanilide, leading to the formation of benzoic acid. The use of microwave irradiation not only speeds up the reaction but also improves the overall efficiency and sustainability of the process by reducing energy consumption and minimizing the generation of by-products.[6]

This modern approach aligns with the principles of green chemistry, aiming to create more sustainable and environmentally friendly chemical processes. Microwave-assisted synthesis is a powerful technique in organic synthesis, particularly for accelerating reactions that traditionally require lengthy heating periods.[7]

MATERIAL AND METHODOLOGY

Microwave Chemistry

Microwave chemistry involves the use of microwave radiation to conduct chemical reactions. Microwaves are a form of electromagnetic radiation with frequencies ranging from 300 MHz to 300 GHz. In chemistry, microwaves typically operate at a frequency of 2.45 GHz. Microwave irradiation provides an efficient and rapid heating method, which can lead to reduced reaction times, higher yields, and cleaner reactions compared to conventional heating methods.[8]

Principles of Microwave Heating

Dielectric Heating

Microwaves interact with polar molecules and ions in a sample, causing them to oscillate rapidly. This oscillation results in friction and heat generation within the sample.

Dielectric heating is dependent on the dielectric constant and loss tangent of the material, which measures the ability to absorb and convert microwave energy into heat.[9]

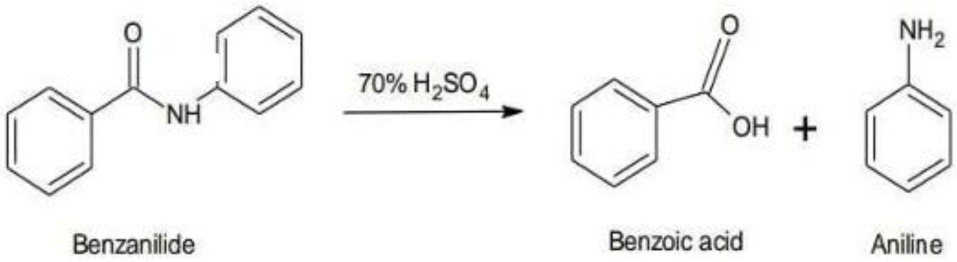
Selective Heating

Microwaves selectively heat materials that absorb microwave energy efficiently. Solvents and reactants with high dielectric constants and loss tangents are heated more effectively.[10]

Reaction Principle

The reaction principle of microwave-assisted creation of benzoic acid from benzanilide when there is sulfuric acid involves the cleavage of the amide bond in benzanilide to form benzoic acid, driven by the heat generated from microwave irradiation and the catalytic action of sulfuric acid. Microwave irradiation accelerates the reaction by rapidly heating the reactants, leading to faster conversion rates compared to conventional heating methods.[11] The sulfuric acid presence likely acts as a catalyst, facilitating the reaction by protonating the amide nitrogen and enhancing the electrophilicity of the carbonyl carbon, thereby promoting the cleavage of the amide bond. This process enables efficient conversion of benzanilide to benzoic acid under milder conditions and shorter response times in comparison to traditional techniques. [12]

Reaction



Procedure synthesis of Bezoic Acid [Microwave Assisted]

Fill a 10 ml concentrated sulfuric acid and 3 gm of benzanilide in 250 ml flask with round bottom.
Give the combination a 10-minute exposure at 225 watts.
A portion of the benzoic acid will harden in the condenser after vaporizing in the steam.
Fill the condenser with 30 milliliters of hot water.
This will cause the benzoic acid to partially dissolve.
Use ice water to cool the flask.
Use a Buchner funnel to filter, then dry it.[13]

Figure No.1: Microwave Oven

Procedure synthesis of Benzoic Acid [Conventional Method]

Place 3 gm of benzanilide and 10 ml concentrated sulfuric acid in flask fitted with reflux condenser and boil gently for 30 min.
A portion of the benzoic acid will solidify in the condenser after vaporizing in the steam.
Pour the 30 milliliters of hot water in condenser.
This will cause the benzoic acid to partially dissolve.
Use ice water to cool the flask.
Use a Buchner funnel to filter, then dry it.[14]

REACTION MECHANISM

The synthesis of benzoic acid from benzanilide using sulfuric acid typically involves a process known as hydrolysis. Here's a general overview of the reaction mechanism:

Protonation of the Carbonyl Group: In the presence of sulfuric acid (H2SO4), the carbonyl oxygen of benzanilide is protonated, forming a resonance-stabilized cationic intermediate.[15]

Attack by Water Molecule: Water molecules in the reaction mixture then attack the electrophilic carbon atom of the protonated carbonyl group. This nucleophilic attack results in the splitting of the C-N bond, resulting in the creation of an ammonium ion and a carboxylic acid intermediate.[16]

Deprotonation: The carboxylic acid intermediate is then deprotonated by water or by another water molecule present in the solution, resulting in the formation of benzoic acid.[17]

The Recovery of Sulfuric Acid: Throughout the reaction, Sulfuric acid is created while serving as a catalyst. The overall reaction can be summarized as follows:

Benzanilide + H2O + H2SO4 → Benzoic Acid + NH4+ + HSO4

This reaction mechanism illustrates the conversion of benzanilide, which contains an amide functional group, into benzoic acid, which contains a carboxylic acid functional group. It is significant to remember that the conditions of the reaction, such as temperature and concentration, can influence the reaction rate and yield. Additionally, safety precautions should be taken when working with concentrated sulfuric acid due to its corrosive nature. [18]

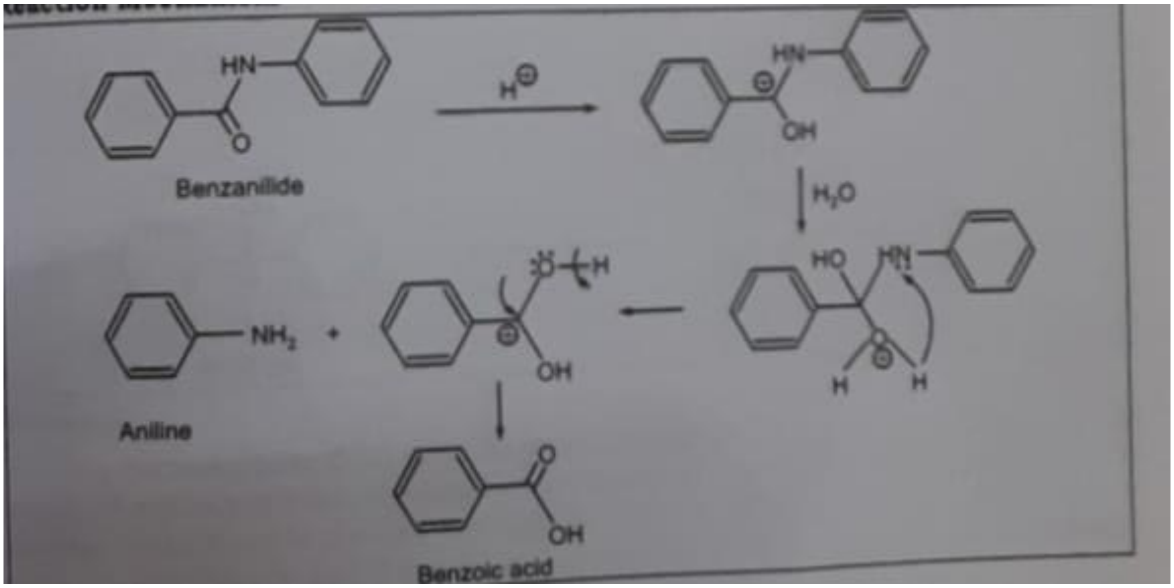


Figure No. 2: Reaction Mechanism of Benzoic Acid

RESULT:

Calculation [microwave assisted]

Theoretical yield

Molecular weight of Benzanilide (reactant) = Molecular weight of Benzoic Acid (product)

C13 H 11NO =12× 13+1×11+14×1+16×1=197 = C7 H 6O2 =12×7+1×6+16×2 =122

No of moles of Benzanilide = mass / molar mass = 3/197 = 0.0152

Theoretical yield = No of moles of of benzanilide × molar mass of benzoic acid

0.0152 × 122 = 1.85 g

Practical yield = 1.56 g

Percentage (%) yield = Practical yield \ Theoretical yield

1.56 / 1.85 = 84.32 %

Theoretical yield = 1.85 g Practical yield = 1.56 g Percentage (%) yield = 84.32 %

Calculation [conventional method]

Theoretical yield = 1.85 g Practical yield = 1.15 g Percentage (%) yield = 1.15 / 1.85 = 62.16 %

Colour	white or colour less
Appearance	White crystalline solid
Odour	Pleasant odour
State	Solid
Melting point	119 OC
PH determination	4.2 PH
Flame Test	burn with smoky flame
Solubility	soluble in ethanol

Table No 1: Physical Properties

Identification tests

Sr No.	Test	Observation	Inference
1	Add 1 milliliter of 1 M NaOH, slowly warm 0.2 g with 20 ml of water, and filter. Add the ferric chloride test solution to the filtrate.	Buff coloured precipitate is produced	Test is positive
2	Dissolve 1 milliliter of sodium bicarbonate in water to get a saturated solution. To the saturated sodium bicarbonate solution, add the specified amount of benzoic acid. Give the mixture a good shake.	Brisk effervescence is observed	Test is positive

Table No.2: Identification test

Limit Test

Sr No.	Test	Inference
1	**Arsenic Test**: Arsenic Combination 5.0 gram with 3 gram of anhydrous sodium carbonate, then fully combine with 10 milliliter of bromine solution. On a water bath, evaporate until dry. Then, use 16 milliliter of brominated hydrochloric acid and 45 milliliters of water to dissolve the cooled residue. Use two milliliters of stannous chloride as to eliminate the excess bromine. The resultant solution passes the arsenic (2 ppm) limit test.	Test is positive
2	**Heavy Metal Test**: - When two milliliters of water and ten milliliter of hydrogen sulphide solution are added to a solution made with twenty-five milliliters of acetone, two milliliter of lead standard solution (10 parts per million Pb), and ten milliliter of hydrogen sulphide solution, no more colour is created.	Test is positive

Table No.: 3: Limit Test

DISCUSSION

Microwave-assisted synthesis is a method where microwave irradiation is used to accelerate chemical reactions. In the case of benzoic acid synthesis, this technique can be advantageous due to its ability to heat reaction mixtures rapidly and uniformly, potentially reducing reaction times and improving yields compared to conventional heating methods. Benzoic acid can be synthesized through various methods, including oxidation of toluene or benzyl chloride, and carboxylation of benzene or its derivatives.

Microwave irradiation can enhance these reactions by increasing molecular collisions and thereby speeding up the formation of benzoic acid.

Microwave heating can significantly reduce reaction times, sometimes from hours to minutes, by efficiently transferring energy to the reaction mixture. Improved heating uniformity often leads to better control over reaction conditions, which can enhance product yields. Despite the rapid heating, microwave synthesis can be more energy-efficient than conventional methods due to shorter reaction times. conditions such as temperature, solvent choice, and microwave power is crucial to maximize these benefits and ensure reproducibility.

CONCLUSION

In this study, microwave-assisted synthesis of benzoic acid from benzanilide using sulfuric acid proved to be an efficient method for the transformation of the initial substance. The reaction proceeded rapidly under microwave irradiation, yielding benzoic acid with high purity and in good yields. The use of sulfuric acid as a catalyst facilitated the hydrolysis of benzanilide to benzoic acid, and the employment of microwave heating provided rapid and uniform heating throughout the reaction mixture. This resulted in shortened reaction times compared to traditional methods and reduced energy consumption. The simplicity and efficiency of this method make it a promising approach for the synthesis of benzoic acid on a larger scale. Further optimization and scale-up studies could enhance its applicability in industrial settings.

The microwave-assisted synthesis of benzoic acid presents a promising method for the efficient conversion of starting materials. This study demonstrated that microwave irradiation significantly accelerates the reaction, leading to higher yields of benzoic acid compared to conventional heating methods. The use of microwave heating allowed for precise temperature control and uniform heating throughout the reaction mixture, resulting in shorter reaction times and higher purity of the final product. Overall, this method offers a rapid, environmentally friendly, and cost-effective route to synthesizing benzoic acid, with potential applications in both laboratory research and industrial production. Further optimization studies could focus on enhancing the reaction conditions to maximize yield and purity, as well as scaling up the synthesis for large-scale production.

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