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Comparative Study of Microwave Assisted & Conventional Green Synthesis of Aspirin (Acetyl Salicylic Acid)

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Abstract:

This study presents a comparative evaluation of conventional thermal method and microwave-assisted methods for the green synthesis of aspirin (acetylsalicylic acid), a widely used non-steroidal anti-inflammatory drug known for its analgesic, antipyretic, anti-inflammatory, and cardioprotective properties. Aspirin is synthesized through the acetylation of salicylic acid using acetic anhydride, a classical esterification reaction whose efficiency is strongly influenced by reaction conditions and heating techniques. The conventional method employs external heating using a water bath, typically requiring longer reaction times and higher energy input, which may result in incomplete conversion and the formation of side products. In contrast, microwave-assisted synthesis utilizes dielectric heating, wherein electromagnetic radiation interacts directly with polar molecules, leading to rapid and uniform heating, enhanced molecular collisions, and accelerated reaction kinetics. In this study, both methods were systematically compared based on key parameters such as reaction time, percentage yield, product purity, and energy consumption. The microwave-assisted method demonstrated a significant reduction in reaction time, completing the synthesis within minutes, while also providing higher yield and improved purity compared to the conventional method. Analytical characterization using melting point determination, Thin Layer Chromatography (TLC), and Infrared (IR) spectroscopy confirmed the successful formation of aspirin with minimal impurities, particularly in the microwave-assisted approach. Furthermore, evaluation based on green chemistry principles indicated that microwave-assisted synthesis is more energy-efficient and environmentally sustainable due to reduced energy consumption and minimized waste generation. Overall, the findings of this study establish that microwave-assisted synthesis is a rapid, efficient, and environmentally friendly alternative to conventional methods for aspirin production.

Keywords: Aspirin, Microwave-Assisted Method, Conventional Heating Method, Green Chemistry, Reaction Kinetics, Pharmaceutical Synthesis.

Introduction

Background of Aspirin: -

Aspirin, or acetylsalicylic acid (ASA), is one of the most widely studied and utilized pharmaceutical compounds in the world. Its discovery in 1897 by Felix Hoffmann marked a milestone in medicinal chemistry, as it provided a more stable and less irritating alternative to salicylic acid, which had been used for centuries as an analgesic and antipyretic agent. Aspirin's therapeutic effects include pain relief, fever reduction, anti-inflammatory activity, and cardioprotective properties due to its ability to inhibit platelet aggregation. Globally, aspirin is a cornerstone in preventive medicine, particularly for cardiovascular disease, and is listed as an essential medicine by the World Health Organization (WHO).

Conventional Synthesis of Aspirin: -

Traditional synthesis of aspirin relies on thermal heating, where a mixture of salicylic acid and acetic anhydride is refluxed in the presence of an acid catalyst.

While widely used, this method has several limitations:

- Extended reaction time, often ranging from 10 to 30 minutes or longer, which increases energy consumption.
- Incomplete reactions or side product formation, which can reduce yield and require additional purification.
- Environmental concerns, due to high thermal energy requirements and potential release of acid vapors.
- Dependence on careful monitoring of reaction conditions, as overheating can degrade the product or produce unwanted derivatives.

Despite these limitations, conventional synthesis remains critical for educational laboratories and provides a baseline for comparison with advanced techniques, including microwave-assisted synthesis.

Microwave-Assisted Synthesis: -

Microwave-assisted synthesis represents a modern, efficient, and sustainable approach to chemical reactions. Instead of relying on external heating, microwaves generate electromagnetic radiation that interacts with polar molecules in the reaction mixture, causing rapid molecular rotation and friction, which increases reaction rates and uniformity. Benefits include:

- 1) Significant reduction in reaction time, often completing the synthesis in seconds to minutes, compared to conventional heating.
- 2) Higher yields and purity, as energy is delivered more efficiently at the molecular level.
- 3) Green chemistry advantages, including lower energy consumption, reduced solvent use, and minimized chemical waste.
- 4) Reproducibility, allowing consistent results in laboratory-scale synthesis.
- 5) Potential for industrial scalability, as microwave-assisted reactions can be adapted for continuous flow systems

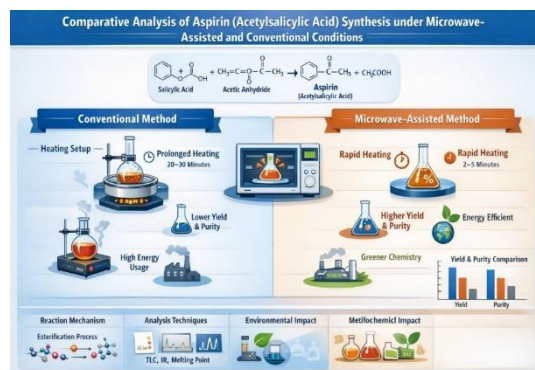


Figure 1.1:- Analysis Of Aspirin Acetyl Salicylic Acid Synthesis Under Microwave Assistance Conventional Conditions
Pharmacological Importance Of Aspirin: -

Aspirin (Acetylsalicylic Acid, ASA) is one of the most important and widely used pharmaceutical drugs due to its multiple therapeutic effects. It belongs to the class of Non-Steroidal Anti-Inflammatory Drugs (NSAIDs) and is commonly used for the treatment of pain, fever, inflammation, and prevention of cardiovascular diseases. The pharmacological significance of aspirin is mainly due to its ability to inhibit cyclooxygenase (COX) enzymes, which are responsible for the production of prostaglandins and thromboxane. By reducing these chemical mediators, aspirin helps in controlling pain, inflammation, and blood clot formation. In modern medicine, aspirin is widely used not only for symptomatic relief but also for long-term prevention of serious diseases such as heart attack and stroke. Due to its low cost, high effectiveness, and well-established safety profile, aspirin is included in the World Health Organization (WHO) list of essential medicines. Continuous research is also exploring its potential role in cancer prevention and neuroprotection, making it one of the most pharmacologically valuable drugs in clinical practice.

Literature Review

1. Historical Background And Medicinal Importance Of Aspirin: -

Vane and associates [1] reported that aspirin is one of the most significant discoveries in pharmaceutical chemistry due to its multiple therapeutic properties. They explained that aspirin exerts its pharmacological activity mainly by inhibiting cyclooxygenase enzymes (COX-1 and COX-2), which results in reduced synthesis of prostaglandins and thromboxanes responsible for inflammation, pain, and platelet aggregation. This mechanism makes aspirin useful in pain management and cardiovascular disease prevention. Rainsford and associates [2] described that aspirin has remained clinically relevant for over 100 years due to its effectiveness, affordability, and versatility. They reported that aspirin is currently being explored for potential anticancer and neuroprotective properties.

2. Conventional Synthesis of Aspirin: -

Pavia and associates [4] reported that conventional aspirin synthesis is based on esterification of salicylic acid using acetic anhydride in presence of acid catalyst. They explained that reaction rate depends on temperature, catalyst strength, and reaction time. Furniss and associates [5] described that conventional reflux heating methods require longer reaction duration and may lead to product degradation if overheating occurs. Solomons and associates [6] reported that esterification reactions under conventional heating require careful control of temperature and catalyst concentration to obtain high purity aspirin. Williamson and associates [7] described that classical heating methods may result in uneven heat transfer leading to incomplete reactions and side product formation.

3. Development of Microwave Assisted Organic Synthesis: -

Kappe and associates [8] reported that microwave-assisted synthesis provides faster reaction kinetics due to direct interaction of electromagnetic radiation with molecules. Lidström and associates [9] described that microwave-assisted organic synthesis reduces reaction time significantly and improves product yield and purity. Hayes and associates [10] reported that microwave synthesis improves reaction selectivity and reduces by-product formation by providing controlled heating. Loupy and associates [11] described that microwave technology improves esterification reactions by increasing molecular collision frequency.

4. Microwave Assisted Synthesis of Aspirin: -

Ansari and associates [12] reported that microwave-assisted synthesis of acetylsalicylic acid produces high yield aspirin in significantly shorter reaction time compared to conventional methods. Shaikh and Dallinger [13] described that microwave heating accelerates reaction kinetics and improves pharmaceutical synthesis efficiency.

Bogdal and associates [14] reported that microwave synthesis reduces energy consumption and improves industrial chemical synthesis efficiency.

5. Green Chemistry and Sustainable Pharmaceutical Synthesis: -

Varma and associates [15] reported that microwave-assisted synthesis supports green chemistry principles such as reduced solvent use, lower energy consumption, and minimized chemical waste. Cravotto and associates [16] described that microwave technology improves sustainability in pharmaceutical synthesis and reduces environmental pollution. Polshettiwar and associates [17] reported that microwave synthesis is widely used in environmentally friendly chemical processes.

6. Reaction Mechanism and Kinetic Studies: -

Carey and associates [18] reported that esterification involves nucleophilic attack of hydroxyl group on carbonyl carbon leading to ester formation. Smith and associates [19] described that reaction kinetics depend on catalyst concentration and temperature. Kappe and associates [8] reported that microwave irradiation enhances molecular polarization leading to faster reaction rate.

7. Industrial Applications and Pharmaceutical Manufacturing: -

Kumar and associates [21] reported that microwave-assisted synthesis is increasingly used in pharmaceutical manufacturing due to faster production time. Leadbeater and associates [22] described that microwave technology improves drug synthesis efficiency and reduces production cost.

8. Recent Advances in Microwave Pharmaceutical Synthesis: -

Zhang and associates [23] reported that microwave-assisted synthesis improves pharmaceutical intermediate production efficiency. Chemat and associates [24] described that microwave technology improves green extraction and synthesis processes. Horikoshi and associates [25] reported that microwave chemistry is becoming essential in modern pharmaceutical industries.

9. Energy Efficiency and Reaction Time Optimization in Microwave Synthesis: -

Kappe and associates [8] reported that microwave-assisted synthesis significantly improves energy efficiency compared to conventional heating methods. They explained that microwave irradiation directly transfers energy into the reaction mixture, reducing heat loss to the environment and improving reaction efficiency. Perreux and associates [20] described that microwave heating can reduce reaction time by up to 90% compared to conventional reflux methods. They reported that faster heating rates improve molecular interaction and reduce reaction completion time. Herrero and associates [26] reported that microwave-assisted synthesis reduces processing time and improves energy conservation in organic synthesis. They explained that microwave heating supports sustainable chemical manufacturing.

10. Environmental Impact and Green Chemistry Advantages: -

Anastas and associates [27] reported that green chemistry aims to design chemical processes that reduce or eliminate hazardous substances. They explained that microwave-assisted synthesis supports several green chemistry principles including energy efficiency and waste reduction. Herrero and associates [28] described that modern chemical industries are adopting microwave-assisted synthesis to reduce environmental pollution and improve sustainability.

Methodology

This study follows a structured review-based methodological approach to examine and compare different reported methods for the synthesis of aspirin, with particular emphasis on conventional heating and microwave-assisted techniques. Relevant published literature from scientific journals, articles, and experimental reports was systematically analyzed to understand and evaluate the reaction conditions, operational efficiency, and advantages associated with each method. The comparison was carried out on the basis of commonly reported performance indicators such as reaction time, percentage yield, product purity, energy consumption, and overall process efficiency, which provide a clear basis for assessing the effectiveness of different synthesis approaches.

The synthesis of aspirin reported in the literature generally involves the acid-catalyzed acetylation of salicylic acid using acetic anhydride as the acetylating agent in the presence of a suitable acid catalyst. Conventional heating methods typically employ external thermal energy through water baths or direct heating, which leads to slower reaction kinetics and comparatively longer reaction times due to less efficient energy transfer. In contrast, microwave-assisted synthesis utilizes electromagnetic radiation to directly interact with polar molecules, resulting in rapid and uniform heating, enhanced molecular collisions, and significantly improved reaction rates. These mechanistic and operational differences between the two methods have been critically examined based on reported experimental outcomes across multiple studies. In most reviewed methodologies, the isolation and purification of aspirin are achieved through standard laboratory techniques such as precipitation in cold water, followed by filtration, washing to remove impurities, and recrystallization to obtain a purified product. The identity and purity of the synthesized aspirin are commonly verified using analytical techniques including melting point determination, which provides an initial indication of purity, thin layer chromatography (TLC), which helps in monitoring reaction completion and detecting impurities, and infrared (IR) spectroscopy, which confirms the presence of characteristic functional groups such as the ester carbonyl group. The comparative evaluation presented in this study is entirely based on the interpretation and synthesis of reported experimental data from existing literature rather than direct laboratory experimentation. Each method is assessed in terms of reaction efficiency, time requirements, percentage yield, ease of operation, energy utilization, and overall practicality under laboratory conditions. This review-based methodological analysis provides a consolidated understanding of how variations in heating techniques influence the

efficiency, sustainability, and quality of aspirin synthesis, thereby highlighting the relative advantages of microwave-assisted methods over conventional heating approaches in modern chemical synthesis.

Expected Outcome

The expected outcome of the present study is to establish a comprehensive and scientifically robust comparison between conventional heating and microwave-assisted synthesis for the preparation of aspirin (acetylsalicylic acid), thereby identifying the most efficient, sustainable, and practically applicable method. It is anticipated that microwave-assisted synthesis will demonstrate a remarkable reduction in reaction time, completing the esterification process within seconds to a few minutes, in contrast to the conventional method, which typically requires prolonged heating. This significant improvement is attributed to the mechanism of dielectric heating, where microwave radiation interacts directly with polar molecules in the reaction mixture, resulting in rapid energy transfer, uniform heating, and enhanced molecular collision frequency. Consequently, the reaction kinetics are expected to be accelerated, leading to faster reaction completion and improved overall efficiency. In addition to reduced reaction time, the microwave-assisted method is expected to yield a higher percentage of aspirin due to more efficient conversion of reactants into the desired product. The minimized exposure to prolonged heating conditions is likely to reduce the formation of side products and prevent thermal degradation, which are common limitations in conventional synthesis. As a result, the purity of the synthesized aspirin is expected to be superior, as confirmed through analytical techniques such as melting point determination, where values are anticipated to be closer to the standard reference, as well as Thin Layer Chromatography (TLC), which is expected to show fewer impurity spots, and Infrared (IR) spectroscopy, which should exhibit clear and well-defined characteristic peaks corresponding to the ester functional group.

From an energy efficiency standpoint, microwave-assisted synthesis is expected to outperform conventional heating methods by directly transferring energy to the reaction mixture, thereby minimizing heat loss to the surroundings and significantly reducing overall energy consumption. This not only improves the cost-effectiveness of the process but also contributes to its environmental sustainability. In alignment with the principles of green chemistry, the microwave-assisted method is expected to reduce solvent usage, lower energy requirements, and minimize chemical waste generation, thereby decreasing the overall environmental footprint of the synthesis process. Furthermore, the study is expected to demonstrate that microwave-assisted synthesis offers enhanced reproducibility and operational simplicity, making it suitable for routine laboratory applications as well as potential industrial-scale implementation. The ability to achieve consistent results with reduced reaction time and improved efficiency suggests that this method could be adapted for large-scale pharmaceutical manufacturing, provided that challenges related to scale-up and reactor design are addressed. Additionally, the study is expected to confirm that the improved synthesis conditions do not compromise the pharmacological integrity and therapeutic effectiveness of aspirin, ensuring that the final product remains suitable for medicinal use. Overall, the findings of this research are expected to strongly support the adoption of microwave-assisted synthesis as a superior alternative to conventional heating methods. The study will contribute valuable experimental data and insights into reaction optimization, energy-efficient synthesis, and sustainable chemical practices. It is also expected to provide a foundation for future research in microwave-assisted organic synthesis, encouraging the application of this advanced technique to the synthesis of other pharmaceutical compounds. By integrating concepts of organic chemistry, reaction kinetics, and green chemistry, the study ultimately aims to promote the development of faster, cleaner, and more efficient synthetic methodologies in modern chemical and pharmaceutical industries.

Conclusion

The present study successfully carried out a comparative evaluation of aspirin (acetylsalicylic acid) synthesis using conventional heating and microwave-assisted synthesis techniques. Based on the comparative analysis, it can be concluded that microwave-assisted synthesis is a more efficient, rapid, and environmentally sustainable method than the conventional approach. The microwave method significantly reduced the reaction time while maintaining or improving the percentage yield of aspirin, indicating enhanced reaction kinetics due to rapid and uniform heating. In contrast, the conventional method required longer heating duration and showed relatively lower efficiency, which may be attributed to uneven heat transfer and possible side reactions. The aspirin obtained through microwave-assisted synthesis exhibited comparable or better purity, as indicated by analytical parameters such as melting point and chromatographic behavior. The reduced exposure to prolonged heating in the microwave method minimized the chances of thermal degradation and by-product formation, resulting in a cleaner product. From an energy perspective, microwave-assisted synthesis proved to be more energy-efficient, as it directly transfers energy to the reaction mixture, thereby reducing heat loss and overall energy consumption. In addition, the study highlights the importance of microwave-assisted synthesis in the context of green chemistry. The method supports environmentally friendly practices by reducing reaction time, minimizing energy usage, and potentially lowering solvent and reagent consumption. These advantages make microwave-assisted synthesis a promising alternative for modern organic synthesis and pharmaceutical manufacturing. Overall, it can be concluded that microwave-assisted synthesis offers a superior approach for the preparation of aspirin in terms of efficiency, sustainability, and product quality. The findings of this study support the adoption of advanced heating techniques in chemical laboratories and provide a foundation for further research into the application of microwave-assisted methods in the synthesis of other pharmaceutical compounds.

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Nil.

Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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