

Review of Recent Developments in Microwave-Assisted Synthesis

Gurumeet Wadhawa², Shubham Nakhawa¹, Nitesh Dhamankar¹,
Maryappa Sonawale², Chinmay Patil¹

Student Department of Chemistry, Veer Wajekar ASC College, Phunde, Uran, Raigad¹
Assistant Professor Post Graduate Department of Chemistry, Veer Wajekar ASC College, Phunde, Uran, Raigad²

Abstract: *Green chemistry is defined as "the discovery, design, and use of chemical products and processes that reduce or eliminate the use and synthesis of hazardous compounds." Regulation, control, and remediation are key applications of green chemistry theory and practice; the environmental benefit can thus represent its economic impact. This also necessitates the replacement of existing heating systems with modern technologies such as microwave radiation, which help to reduce the carbon footprint to the greatest extent possible.*

I. INTRODUCTION

Green chemistry requires microwave synthesis. Microwave radiation, a kind of electromagnetic radiation, is extensively utilized in chemical synthesis as a heat source. As a "leader" in organic synthesis, the development of microwave assisted organic synthesis boosted green chemistry. This approach has inspired several chemists to research microwave assisted chemistry. The use of microwaves as an energy source for controlling synthetic reactions has advanced dramatically. Obviously, microwave synthesis is a technique that allows laboratory chemists to achieve their objectives in a fraction of the time that standard heat transfer procedures require. [1-5] this page explains microwave synthesis, its manufacturing technique, and the most recent advancements in this sector. Microwaves' widespread availability has increased their usage in chemistry, and microwave heating has evolved into a formidable instrument for speeding a wide range of chemical reactions. 3. Optimization, or finding the best conditions for a certain reaction to produce the desired products in high yields and purity, is frequently the bottleneck of classical synthesis. Because many synthesis reactions entail one or more lengthy heating steps, optimizing them is frequently challenging and time-consuming. Microwave heating under controlled conditions has proven to be an appealing technology for any application involving the heating of a reaction mixture, as it frequently reduces reaction times from days or hours to minutes or even seconds. [6-9] this groundbreaking novel technology allows for the quick parallel or sequential (automated) synthesis of compounds. Robert Bunsen invented the hot plate in 1899 while studying organic chemistry. This significant development allows for considerably more targeted heat generation for chemical synthesis. 4 The Bunsen burner was eventually superseded by oil baths or hotplates, and microwave energy technology became important in science in the twenty. [10-14] first century.

The dynamic temperature range given by current laboratory equipment is appealing in terms of chemical synthesis. The great majority of studies have concentrated on high temperature transformations or heating dependent reactions. Microwaves, on the other hand, have recently been utilized to effect synthetic alterations that would be impossible to achieve through traditional heating. Microwave technology advancements have enabled low temperature reactions to occur within the parameters required for biological applications. Wavelengths in the electromagnetic spectrum range from 1 cm to 1 m, while microwave frequencies range from 30 GHz to 300 MHz's this place it between the radio and infrared spectrums. The frequency bands 91515 MHz and 245050 MHz are industry standard. [15-16] the shorter wavelength is used for radar and the remaining wavelength is used for communications.

As a result, heating applications cannot use the entire microwave spectrum, and devices operating at 2.45 GHz are the standard. Magnetic and electric forces are present in microwave energy. Microwave energy differs from other types of radiation in that it is nonionizing and hence does not modify the molecular structure of the heated substances; instead, it offers thermal activation. [16-18] the heating effect used in microwave assisted organic conversions is mostly due to dielectric polarization.

A fast-fluctuating electric field (2.45×10^9 Hz) strikes the molecule, causing it to continuously absorb energy as it tries to cope with the fluctuating field. The dielectric constant influences a material's ability to convert electromagnetic energy into thermal energy. Microwave coupling increases as the dielectric constant increases. Microwaves easily heat solvents such as water, methanol, DMF, ethyl acetate, acetone, and acetic acid. When subjected to microwave radiation, low dielectric constant solvents such as hexane, toluene, carbon chloride, and others do not condense and so do not heat up as quickly. Microwave heating has shown to be an extremely useful source of heat not just in the kitchen, but also in the laboratory. Chemists studied whether it was possible to use a normal microwave oven for chemical procedures. Yet, since the mid-1980s, the benefits of utilizing microwave dielectric heating for organic transformations⁷ have been abundantly apparent. This approach enables synthetic chemists to carry out previously difficult to perform reactions using conventional heating. Green chemistry was thus guided by atomic economy and energy considerations.

As defined in the "12 Principles of Green Chemistry," these are waste control, atomic economy, less hazardous chemical synthesis, and the discovery of safer compounds. Solvent and additive modification. Microwave dielectric heating is obviously employed for organic transformations, according to a proposal for energy efficiency and the utilization of renewable raw materials. This approach enables synthetic chemists to perform previously difficult reactions using standard heating. As a result, the ideas of atomic economy and energy issues became the driving forces behind green chemistry. The "12 principles of green chemistry" are waste prevention, atomic economy, less harmful chemical synthesis, and the creation of safer molecules. Solvents and additives are being improved. Employ sustainable basic materials; Utilize fewer derivatives; and Design for energy efficiency. [19-21]

1.1 Microwave Reaction Principles

Microwaves' widespread availability has increased their usage in chemistry, and microwave heating has evolved into a formidable instrument for speeding a wide range of chemical reactions³. Optimization, or finding the best conditions for a certain reaction to produce the desired products in high yields and purity, is frequently the bottleneck of classical synthesis. Because many synthesis reactions entail one or more lengthy heating steps, optimizing them is frequently challenging and time-consuming. Microwave heating under controlled conditions has proven to be an appealing technology for any application involving the heating of a reaction mixture, as it frequently reduces reaction times from days or hours to minutes or even seconds. This groundbreaking novel technology allows for the quick parallel or sequential (automated) synthesis of compounds. Robert Bunsen invented the hot plate in 1899 while studying organic chemistry. This significant development allows for considerably more targeted heat generation for chemical synthesis. The Bunsen burner was eventually superseded by oil baths or hotplates, and microwave energy technology became important in science in the twenty-first century⁵.

The dynamic temperature range given by current laboratory equipment is appealing in terms of chemical synthesis. The great majority of studies have concentrated on high temperature transformations or heating dependent reactions. Microwaves, on the other hand, have recently been utilized to effect synthetic alterations that would be impossible to achieve through traditional heating. Microwave technology advancements have enabled low temperature reactions to occur within the parameters required for biological applications. Wavelengths in the electromagnetic spectrum range from 1 cm to 1 m, while microwave frequencies range from 30 GHz to 300 MHz's this place it between the radio and infrared spectrums. The frequency bands 91515 MHz and 245050 MHz are industry standard. The shorter wavelength is used for radar, and the remaining wavelength is used for communications. As a result, heating applications cannot use the entire microwave spectrum, and devices operating at 2.45 GHz are the standard. Magnetic and electric forces are present in microwave energy. Microwave energy is distinguished from other types of radiation by its shorter wavelength than X-rays and X-rays. [22-25]

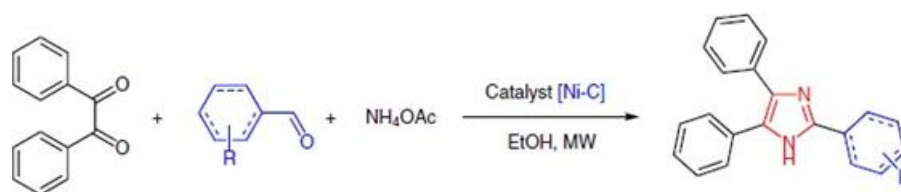
II. APPLICATIONS OF SYNTHESIS

Microwave energy has numerous commercial applications. and minerals have been conducted. Microwaves have been researched for application in chemical synthesis and processing, as well as waste treatment, in addition to plasma processes (surface treatment, chemical vapour infiltration, and powder processing). Despite great effort in developing microwave technology, the majority of microwave research remains in the laboratory.

Numerous recent researches have uncovered a slew of novel compounds produced by microwave irradiation. This section will go over numerous synthetic applications. Roshni V and colleagues used microwave pyrolysis to create NdopedC.dots from sesame seeds. The fluorescence properties and photostability of C.dot are studied. These are C. To comprehend the features of Fe₃metaldetection +, dots will be employed. Microwave heating was utilised to make C.dots from sesame seeds in a time and energy efficient manner that was also environmentally friendly, affordable, and made use of recycled trash. To be commercially viable for industrial usage, zeolites must be produced fast and cheaply. Umer Khalil et al. created a zone of pure mordenite (MOR) zeolite using microwave irradiation. By optimising synthesis parameters such as crystallisation duration, ageing length, and Si/Al ratio, phase purity, crystallinity, and shape were thoroughly examined. Calcium phosphate-based biomaterials are frequently employed as bone substitutes and osteoconductive scaffolds due to their chemical proximity to the inorganic component of bone.

The most prevalent calcium phosphate biomaterial is hydroxyapatite (HA), which can be produced naturally or synthetically. It has been proposed that nanoHA (nHA) particles exhibit increased bio resorption and a chemical and crystallographic structure akin to native bone apatite. Hassan and colleagues proved that microwave (MW) processing of biomaterials, particularly bio ceramics, has advantages over traditional heating methods. Unlike traditional heating, it generates heat within the material's molecules rather than using an external heating source and subsequent radiative transmission. [26]

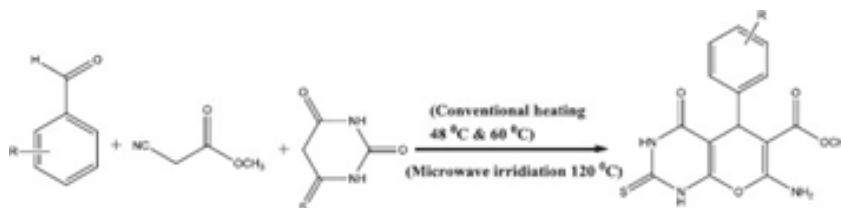
pot synthesis of 2,4,5trisubstituted imidazoles from aldehydes, benzyl, and ammonium acetate. Simple filtration could be employed to recover and reuse the catalyst. According to the findings of twenty researchers, microwave irradiation enhanced yield compared to traditional heating. Overall, using aromatic aldehydes, benzyl, and ammonium acetate as nitrogen sources, this strategy provides a direct and efficient method for producing imidazole derivatives. Researchers created an efficient adsorbent, ironmodified activated carbon fibre (Fe₂O₃/ACF), to extract as (V) from simulated wastewater. Ms. Hasanpoora M. Using a microwave hydrothermal technique and colleagues produced zinc oxide nanoparticles with various morphologies. The hydrothermal reaction conditions govern the size and structure of the zinc oxide particles in this procedure, whereas chemical processes frequently require too long a reaction time. Ajmal R. Bhata and colleagues suggested a microwave, one-pot, three component condensation process with water as the greensolventmethyl-7-amino-4-oxo-5-phenyl-2-thioxo-2,3,4,5-tetrahydro-1H-pyran[2,3-d]pyrimidine -6-carboxylates. Microwave radiation was employed by scientists to synthesise methyl-7-amino-4-oxo-5-phenyl-2-'thioxo-2,3,4,5-tetrahydro-1H-Pyran[2,3-d] pyrimidine-6-carboxylate derivatives.



A comparable impact was previously documented under typical thermal conditions, monitored (48 and 60 degrees Celsius). Under microwave irradiation, the reaction was completed in 3-6 minutes with an excellent yield (78.94%), whereas conventional heating produced moderate yields (69-86%) in 2-6 hours at 48 °C and (71 87%) in 14 hours at 60 °C. Furthermore, yields of selected compounds (67-82%) were produced in 2-7 hours at room temperature. The invention of a precise method for synthesising cobamamide (CN)₂Cbi, a precursor to vitamin B12, will almost certainly increase its cost. (CN)₂Cbi is the most useful product of heating vitamin B12 with NaCN in a microwave reactor. When the purification procedure was simplified, 94% of the product was easily separated. Microwave heating assisted in the synthesis of (CN) ₂Cbi (c-lactone). (CN) ₂Cbi was produced by reacting (CN) ₂Cbi with triethanolamine (c-lactam) [25] Microwave irradiation is an entirely new field of study in polymer science. This type of heating can speed up the reaction and embellish the precise dwellings of the polymer.

Nassima Mazouzi Sennour and colleagues discussed a study that looked at the influence of microwave (MW) selective heating on the polyesterification reaction. The majority and aqueous emulsion polyesterization of sebacic acid with decanediol were investigated as model techniques. Dodecylbenzenesulfonic acid (DBSA), which functions as both a

catalyst and a surfactant, catalyzed the procedure. MW heating creates polyester with a higher molecular weight than standard heating in both bulk and aqueous environments. A microwave assisted combustion process (MWAC) was developed to create Nano crystalline ZnO powder by employing zinc nitrate (as the oxidant) and urea (as the gasoline) as the starting components and water as the solvent, followed by heating the resulting solution in a microwave oven. Studies by L.C.Nehru and others According to colleagues, microwave heating may be used to produce similarly porous ZnO in a matter of minutes. What materials are incompatible with microwaves Very exothermic reactions are no longer permitted in the system.



At high temperatures, explosive hydrogen peroxide cannot be employed, regardless of the method. Because of the closed vessel device, extra precautions must be taken while running with ion rich or gasoline releasing reaction mixtures, as the heating price and pressure rise can be noticeably rapid. At those conditions (extremely diluted solution), the test can be completed with little awareness.

III. CONCLUSIONS

Microwave assisted methods have gained popularity in the domains of polymer science, bioinformatics, biotechnology, and biochemistry in recent years. Microwave ovens can be used to successfully perform chemical processes. This system can effectively provide rapid lead creation and optimization. In general, microwave heating is unquestionably advantageous for organic and medicinal chemists. For heat dependent synthetic transformations, this enabling technology has progressed from a "last option" to a "preferred strategy." "Microwave synthesizers will become an intrinsic component and standard technology in the great majority of synthesis laboratories in the future, and they will continue to benefit organic synthesis, materials manufacture, and biomaterials synthesis. Because of its efficiency and potential to assist develop ecologically friendly products, microwave technology offers interesting applications.

REFERENCES

- [1]. Kappe C. O., Dallinger D., The impact of microwave synthesis on drug discovery. *Nat. Rev. Drug Discovery*, 5: 51-63, 2006.
- [2]. Kappe C. O., Dallinger D., Murphree S. S., Practical microwave synthesis for organic chemists -Strategies, instruments, and protocols. 1st Ed. Wiley-VCH, Verlag GmbH & Co. KGaA, Weinheim, 2009.
- [3]. Gedye, R.; Smith, F.; Westaway, K.; Ali, H.; Baldisera, L.; Laberge, L. and Roussel, J. The use of microwave ovens for rapid organic synthesis. *Tetrahedron Lett.* 1986, 27, 279-282.
- [4]. SuratiMadhvi, JauhariSmita, Desai K.R. A brief review on Microwave assisted organic reactions *Arch. Appl. Sci. Res.* 2012;4 (1):645-661.
- [5]. N. Leadbeater, *Chemistry World* 2004, 1, 38; (b) D. Adam, *Nature* 2003, 421, 571; (c) V. Marx, *Chem. & Eng. News* 2004, 82, 14; (d) A. Yarnell, *Chem. & Eng. News* 2007, 85, 32.
- [6]. Heravi M M, Ajami D, Mojtahedi M M & Ghassemzadeh M, *Tetrahedron Lett*, 40 (1999) 561. Abramovich R A, *Org Prep Proceed Int*, 23 (1991) 638.
- [7]. Adam, D. (2003) *Nature*, 421, 571-572. (b) Blackwell, H. E. (2003) *Org. Biomol. Chem.* 1, 1251-55.
- [8]. Sharma, S. V.; Rama-sarma, G. V. S.; Suresh, B. (2002), *Indian. J. Pharm. Scien.* 64, 337-344.
- [9]. Johansson, H. (2001) *Am. Lab.* 33, 10, 28-32.
- [10]. Bradley, D. (2001) *Modern Drug Discovery* 4, 32-36.

- [11]. Nitin A. Mirgane, Vitthal S. Shivankar, Sandip B. Kotwal, Gurumeet C. Wadhawa, Maryappa C. Sonawale, Degradation of dyes using biologically synthesized zinc oxide nanoparticles, *Materials Today: Proceedings*, Volume 37, Part 2021, Pages 849-853, ISSN 2214-7853, <https://doi.org/10.1016/j.matpr.2020.06.037>.
- [12]. Nitin A. Mirgane, Vitthal S. Shivankar, Sandip B. Kotwal, Gurumeet C. Wadhawa, Maryappa C. Sonawale, Waste pericarp of ananas comosus in green synthesis zinc oxide nanoparticles and their application in waste water treatment, *Materials Today: Proceedings*, Volume 37, Part 2, 2021, Pages 886-889, ISSN 2214-7853, <https://doi.org/10.1016/j.matpr.2020.06.045>.
- [13]. Shubhada S. Nayak, Nitin A. Mirgane, Vitthal S. Shivankar, Kisan B. Pathade, Gurumeet C. Wadhawa, Adsorption of methylene blue dye over activated charcoal from the fruit peel of plant *Hydnocarpus pentandra*, *Materials Today: Proceedings*, Volume 37, Part 2, 2021, Pages 2302-2305, ISSN 2214-7853, <https://doi.org/10.1016/j.matpr.2020.07.728>.
- [14]. Patil, D.D.; Mhaske, K.D.; Wadhawa, C.G., Antibacterial and Antioxidant study of *Ocimum basilicum* Labiatae (sweet basil), *Journal of Advanced Pharmacy Education & Research* (2011) 2, 104-112.
- [15]. Dinanath PD, Gurumeet WC, 2013. Antibacterial, antioxidant and anti-inflammatory studies of leaves and roots of *Solanum xanthocarpum*. *Unique J Ayurvedic Herb Med* (2013) ;(3):59-63.
- [16]. Dynashwar K. Mhaske, Dinanath D. Patil, Gurumeet C. Wadhawa. Antimicrobial activity of methanolic extract from rhizome and roots of *Valerianawallichii*. *International Journal on Pharmaceutical and Biomedical Research*, 2011; 2(4):107- 111
- [17]. Patil DD, Mhaske DK, Gurumeet MP, Wadhawa C. Antibacterial and antioxidant, anti-inflammatory study of leaves and bark of *Cassia fistula*. *Int J Pharm* 2012; 2(1):401-405.
- [18]. G. C. Wadhawa, M. A. Patare, D. D. Patil and D. K. Mhaske, Antibacterial, antioxidant and anti-inflammatory studies of leaves and roots of *Anthocephalus kadamba*. *Universal Journal of Pharmacy*, 2013.
- [19]. Shubhada S. Nayak, Nitin A. Mirgane, Vitthal S. Shivankar, Kisan B. Pathade, Gurumeet C. Wadhawa, Degradation of the industrial dye using the nanoparticles synthesized from flowers of plant *Ceropegia attenuata*, *Materials Today: Proceedings*, Volume 37, Part 2, 2021, Pages 2427-2431, ISSN 2214-7853, <https://doi.org/10.1016/j.matpr.2020.08.274>.
- [20]. G. C. Wadhawa, V. S. S. Shivankar, Y. A. Gaikwad, B. L. Ingale, B. R. Sharma, S. S. Hande, C. H. Gill and L. V. Gavali, *Eur. J. Pharm. Med. Res.*, 3, 556 (2016).
- [21]. Clark, J.H.; Tavener, S.J. *Alternative Solvents: Shades of Green. Org. Process Res. Dev.* 2007, 11, 149–155. [CrossRef]
- [22]. Procopio, A.; Dalpozzo, R.; De Nino, A.; Maiuolo, L.; Nardi, M.; Romeo, G. Mild and efficient method for the cleavage of benzylidene acetals by using erbium (III) triflate. *Org. Biomol. Chem.* 2005, 3, 4129–4133. [CrossRef] [PubMed]
- [23]. Procopio, A.; Dalpozzo, R.; De Nino, A.; Nardi, M.; Oliverio, M.; Russo, B. Er(OTf)₃ as new efficient catalyst for the stereoselective synthesis of C-pseudoglycals. *Synthesis* 2006, 15, 2608–2612. [CrossRef] 28. Procopio, A.; De Luca, G.; Nardi, M.; Oliverio, M.; Paonessa, R. General MW-assisted grafting of MCM-41: Study of the dependence on time dielectric heating and solvent. *Green Chem.* 2009, 11, 770–773. [CrossRef]
- [24]. Procopio, A.; Cravotto, G.; Oliverio, M.; Costanzo, P.; Nardi, M.; Paonessa, R. An Eco-Sustainable Erbium (III)-Catalysed Method for Formation/Cleavage of O-tert-butoxy carbonates. *Green Chem.* 2011, 13, 436–443. [CrossRef]
- [25]. S. S. Nayak, N. A. Mirgane, K. B. Pathade, V. S. Shivankar and G. C. Wadhawa. “Phytochemical analysis, antioxidant and anti-inflammatory activity of leaves and bark of *Ceropegia rollae* Hemadri,” *Plant Sci. Today*, 8(3), 2021, 425–428. <https://doi.org/10.14719/pst.2021.8.3.906>