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SYNTHESIS OF DIFFERENT OXADIAZOLES: A CRITICAL REVIEW

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ABSTRACT:

Oxadiazoles are an interesting class of five-membered heterocyclic compounds containing two atoms of nitrogen and one atom of oxygen [1]. Their lipophilicity is a key property that affects a drug's ability to arrive at the focus by trans-membrane diffusion, and their action against benign tuberculosis by inhibiting lipid biosynthesis is highly promising. This review was emphasized on the synthesis of different oxadiazoles with scheme. For the same, a detailed literature survey was done. Antimicrobial, anticonvulsant, antiepileptic, antiallergic, anticancer, anti-tubercular, and insecticidal actions are just few of the many biological effects shown by 1,3,4-oxadiazole derivatives. The Green Chemistry Program was launched in 1991 by the Environmental Protection Agency and the National Science Foundation. A joint effort between J.C. To lessen or do away with chemical dangers and environmental degradation, Warner has developed twelve key principles of green chemistry. It is necessary to create new medications for the treatment of pain and inflammation without the frequent adverse effects of NSAIDs (gas, bloating, heartburn, ulcer, stomach discomfort). In conclusion, the oxadiazole derivatives are more effective than nonsteroidal anti-inflammatory drugs (NSAIDs) in treating pain and inflammation-induced arthritis. The compounds of 1,3,4-oxadiazole were synthesized using microwave assistance technique.

Keywords: Synthesis, oxadiazole, benzoxazole, benzothiazole, benzimidazole.

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INTRODUCTION

Oxadiazoles are an interesting class of five-membered heterocyclic compounds containing two atoms of nitrogen and one atom of oxygen [1]. Their lipophilicity is a key property that affects a drug's ability to arrive at the focus by trans-membrane diffusion, and their action against benign tuberculosis by inhibiting lipid biosynthesis is highly promising [2]. The 1,2,3-, 1,2,4-, 1,2,5-, and 1,3,4-oxadiazoles are the four regioisomers that can be found in nature. The 1,2,4- and 1,3,4-isomers are substantially more common in published works than the 1,2,5-isomer, which has a distinct orientation of the side chains (R 1 and R 2). Instead, 1,2,3-oxadizole cycles are extremely brittle; these compounds are typically found in the diazoketone tautomer and are easy to produce [3]. It is worth noting that 1,2,4- and 1,3,4-oxadiazoles are both aromatic compounds because they both satisfy the Huckel rule. However, while 1,3,4-oxadiazole derivatives have much greater aromaticity, 1,2,4-isomers are the least aromatic five-membered heterocyclic systems and are better classified as conjugated dienes [4].

However, if electron-releasing groups are used to substitute the oxadiazole ring, electrophiles will attack at the nitrogen instead. In most cases, the oxadiazole ring will not be broken by a nucleophile [5,6]. However, nucleophilic substitution occurs when halogen atoms are replaced by nucleophiles in halogen-substituted oxadiazole. Nucleophilic substitution occurs at the sp2 carbon of oxadiazole in a manner analogous to that of aliphatic carbons [7].

To get more effective anti-inflammatory and analgesic medicines with fewer or no adverse effects (ulcerogenicity), a novel series of 2-[3-(4-bromophenyl)propan-3-one]-5- -(substituted phenyl)-1,3,4-oxadiazoles (4a-n) was synthesized from 3-(4-bromobenzoyl)propionic acid. The compounds in question were obtained by reacting compound 3 with various aryl acid hydrazides (2a-n) in phosphorous oxychloride [8]. The synthesized compounds' structures were confirmed by infrared spectroscopy, proton nuclear magnetic resonance, and mass spectrometry. The activities of the chemicals named in the title were tested for inflammation reduction, pain relief, ulcer formation, and bacteria killing. Minimum inhibitory concentration (MIC) values were used to quantify antibacterial efficacy. Several compounds demonstrated impressive antibacterial activity, while several others were discovered to have potent anti-inflammatory and analgesic properties [9]. Very little ulcerogenic activity was observed in the newly synthesized drugs. The results of the current study show that the anti-inflammatory and analgesic actions as well as the reduction in ulcerogenic activity were greatly enhanced by cyclizing the carboxylic group of 3 into a new 1,3,4-oxadiazole nucleus [10].

Cancer remains a global health concern in both developed and less developed nations. The Food and Drug Administration has approved a number of anticancer medications for use in treating various malignancies. The majority of them have a structure similar to that of heterocyclic rings. Due to its therapeutic characteristics, the oxadiazole ring system occupies

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a prominent position among heterocycles. There are three structural isomers of this ring system: 1,2,4-oxadiazole, 1,3,4-oxadiazole, and 1,2,3-oxadiazole. Creating novel heterocycles with the ability to tailor their intensity to a variety of organic targets continues to be an appealing, sensible endeavor. Since oxadiazole rings are relatively more stable in biological media, they are frequently used as a recurring theme in the structure of drug candidates [11], often serving as bioisosteric substitutions for carbonyl-containing functional groups like esters, amides, carbamates, and hydroxamic esters. Two carbons, two nitrogens, and one oxygen make up the five-membered aromatic heterocycle known as an oxadiazole. These have recently garnered attention from a variety of research groups due to the fact that they can serve as suitable bioisosteric trade alternatives for certain, carbonyl-containing functional groups [12]. Research articles and patent filings related to heterocycles with an oxadiazole ring continue to rise, indicating a high degree of interest among experts. The 1,3,4-oxadiazole is a great bioisostere of amide and ester functional groups; as a five-membered heterocycle, it is thought to contribute important physicochemical properties and a lot to the bond-making process because it is interested in hydrogen-bonding interactions with various receptors. Heterocyclic compounds with nitrogen and oxygen atoms (1,3,4-oxadiazoles) are starting to seem like important pharmacophores in the pharmaceutical industry. Antimicrobial, anticonvulsant, antiepileptic, antiallergic, anticancer, anti-tubercular, and insecticidal actions are just few of the many biological effects shown by 1,3,4-oxadiazole derivatives. One of the most important tactics for the, designing of potent anti-tubercular medicines is to construct inhibitors of mycobacterial cell-divider biosynthesis, and the 1,3,4-oxadiazole-2-(3H)- thiones have been shown to be effective against Mycobacterium TB H 37 Rv in vitro. Mycobacteria display an array of complex unsaturated lipids in their cell walls including mycocerosic corrosive, mycolic corrosive, arabinogalactans, and peptidoglycans [13].

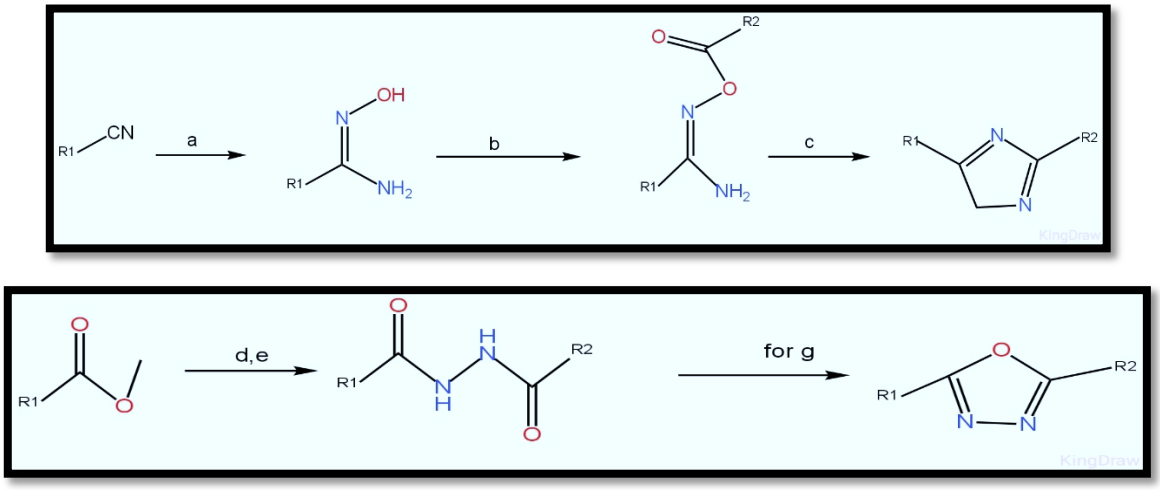

Fig 1. Synthesis scheme of 1,2,4-oxadiazole

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Nitriles (4) and other amidoxime precursors are the most common starting materials for making 1,2,4-oxadiazoles. To complete cyclization following acylation of 5 to the O-acylamidoxime 6, heating at ca. One of the most frequent building blocks for the synthesis of 1,3, 4-oxadiazoles (10) are hydrazide derivatives (9) which can be cyclized at temperatures as low as 100 °C or in the presence of coupling reagents (18). Activation of the monoacylhydrazide moiety, followed by cyclization, gives the 1,3,4-oxadiazole (10), but most preparation methods require strong acidic conditions at elevated temperatures, limiting the practical accessibility of derivatized 1,3,4-oxadiazoles. Milder reaction conditions can be achieved via the generation of phosphonium intermediates or by using 2-chloro-1,3-dimethylimidazolinium chloride (DMC). However, such procedures typically call for more time to complete. While it would be ideal to manufacture a wide range of oxadiazoles at low temperature and fast reaction times to maximize access to the 1,3,4-isomers, none of the existing techniques were deemed robust enough to do so. Phosphonium agents are frequently employed to kick off the dehydration process due to their oxophilicity. Their method tolerates the presence of protective groups which are stable under mild acidic conditions, such as esters and carbamates, and showed excellent stereoscopic control. This method is limited depending on the substitution pattern [14].

Green chemistry- methods

The Green Chemistry Program was launched in 1991 by the Environmental Protection Agency and the National Science Foundation. A joint effort between J.C. To lessen or do away with chemical dangers and environmental degradation, Warner has developed twelve key principles of green chemistry [15].

Microwave irradiation (MWI), ultrasonication, photo-catalysis, grinding, and milling are all examples of green chemistry techniques that can be used to carry out a variety of chemical reactions. The use of these technologies increases the rate of reaction, shortens the reaction time, and increases the product yield, making organic processes more efficient and cost-effective. Mechanochemistry is used in synthetic methods like grinding or milling to create diverse biologically active chemicals quickly, cleanly, efficiently, and solvent-free [16].

- The synthesis must be carried out in such a way that waste or by-product generation is minimized or avoided entirely.
- Atomic economy refers to the practice of developing synthetic procedures that make the most efficient use of starting materials and reagents.
- Use of safer, less toxic alternatives: better design of synthetic processes is needed to ensure that the creation and use of chemicals have negligible or no negative impact on human health and the environment.
- Chemicals can be made safer by careful design, which prioritizes user safety without sacrificing effectiveness.

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- Choose less hazardous solvents, if at all possible, no auxiliary ingredients (solvents, extractants) should be used.
- Low energy consumption is achieved by performing synthesis at room temperature and pressure.
- Raw resources should be renewable as a feedstock.
- Decrease the number of derivatives used; whenever possible, avoid derivatization.
- Catalysis that is selective rather than general is more advantageous than stoichiometric reactions.
- Products made from chemicals should be designed so that they break down into harmless substances when discarded (i.e., they should be biodegradable).
- Monitoring processes in real time to prevent excursions that could result in the creation of harmful compounds by use of real-time analysis.
- Materials used in a chemical process should be chosen to reduce the potential for accidents and harm.

As stated in the first principle of the Rio Declaration on Environment and Development, "Human beings are at the centre of concerns for sustainable developments-they are entitled to a healthy and productive life in harmony with nature" 1, which highlighted the challenge to all of us to define the objectives of sustainable development and to provide scientific, technological, and social tools to achieve those objectives. If we want our next generation of chemicals, materials, and energy to be more sustainable than the current one, chemistry will play a crucial part in making that happen. Green Chemistry, or the application of a set of principles to minimize or eliminate the creation and release of hazardous substances in the production and use of chemicals, is one of the most promising ideas in sustainable chemistry. Green Chemistry has progressed quickly since people have realized that eco-friendly options will save money in the long run. The elimination of solvents or the substitution of ecologically benign solvents in chemical processes is a major part of Green Chemistry. Water, fluorous and ionic liquids, supercritical media, and various combinations thereof are only some of the alternative solvents that are seeing increased use. Because it provides atom-economical, selective, and energy-efficient answers to many significant industrial challenges, catalysis continues to be one of Green Chemistry's most important disciplines. Increased selectivity, softer reaction conditions, and concomitant ease of manipulation have drawn attention to the use of inorganic solid supported reagents. Researchers are interested in microwave heating because it can drastically reduce the time required for reactions, improve reaction selectivity, and boost product yields—all of which are particularly useful when dealing with time-consuming processes that require high temperatures. Large collections or libraries of different heterocycles are commonly used in high-throughput screening throughout the early phases of drug discovery programs due to the drug-like properties and great variety of structural variability.

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This review was based on the synthesis procedures of the followings-

- ✓ 1,2,4-Triazoles
- ✓ Imidazoles and Benzimidazoles
- ✓ Heterocyclic compounds with nitrogen and oxygen atoms
- ✓ 1,2,4-Oxadiazoles and 1,3,4-Oxadiazoles
- ✓ Benzothiazoles

❖ 1,2,4-TRIAZOLES

One-step preparation of 4,5-disubstituted-1,2,4-triazole-3-thiones has been achieved by reacting acid hydrazide with alkyl or aryl isothiocyanate on silica gel and montmorillonite K10 in the presence of a KOH (10%) solution and subjecting the mixture to microwave irradiation. In another method, 4-substituted-1-aroyl thiosemicarbazides are microwave-reacted with a KOH (10%) solution over a silica gel surface to produce the corresponding triazoles **(Fig 2.)** [17].

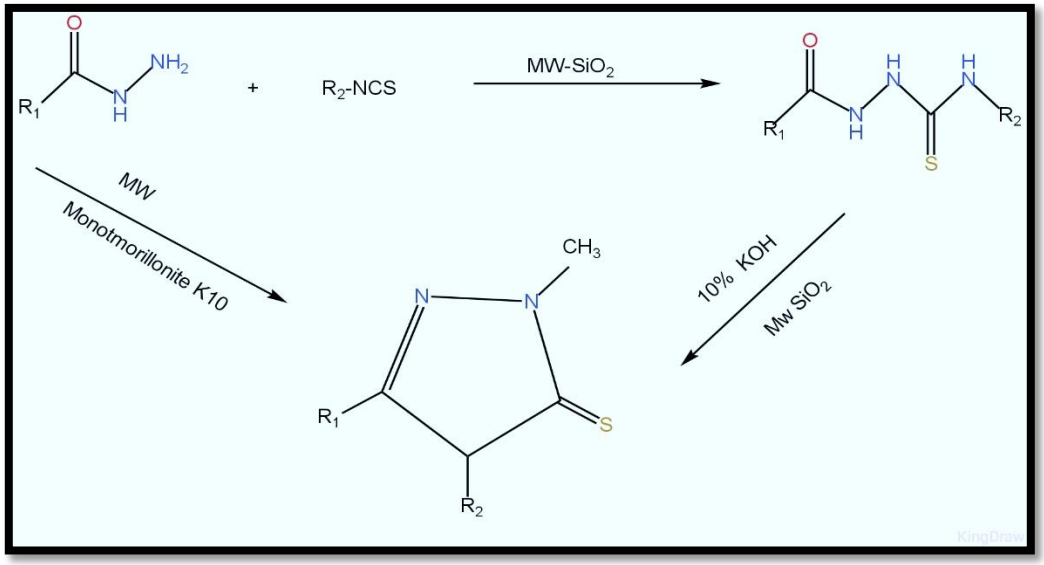

Fig 2.

Microwave irradiation and conventional methods have both been used to produce 4,5-disubstituted 1,2,4-triazole-3-thiones, as reported by Zamani and Bagheri13. Microwave irradiation is discussed for its positive influence on the dehydrative cyclization of thiosemicarbazides in various reaction conditions. Shortening reaction durations (from 2-9 h to 2-4 min) and a little drop (1-4%) in yields were found to be the effects of microwave irradiation on the reaction under study (**Fig 3.)** [18].

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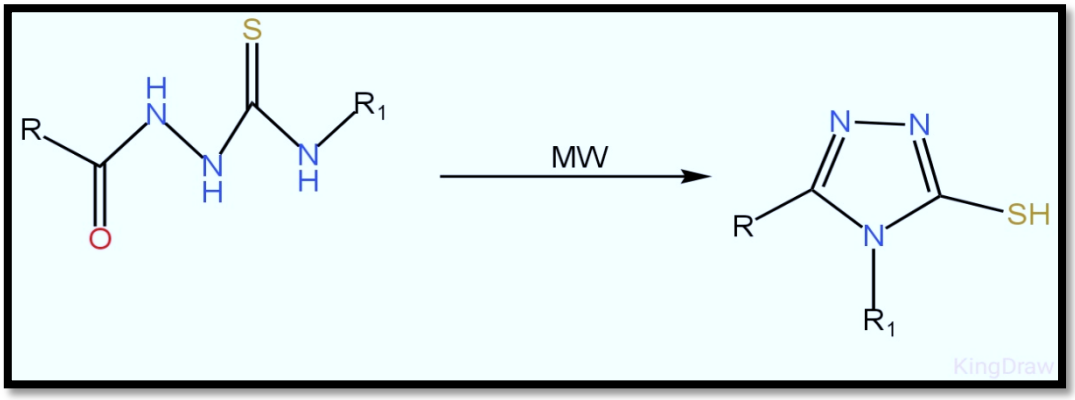

Fig 3.

By reacting aromatic nitriles with an excess of NH2NH22HCl in microwave-heated ethylene glycol containing NH2NH22H2O, Bentiss et al.14 successfully synthesized 3,5-disubstituted-4-amino-1,2,4-triazoles (**Fig 4.).**

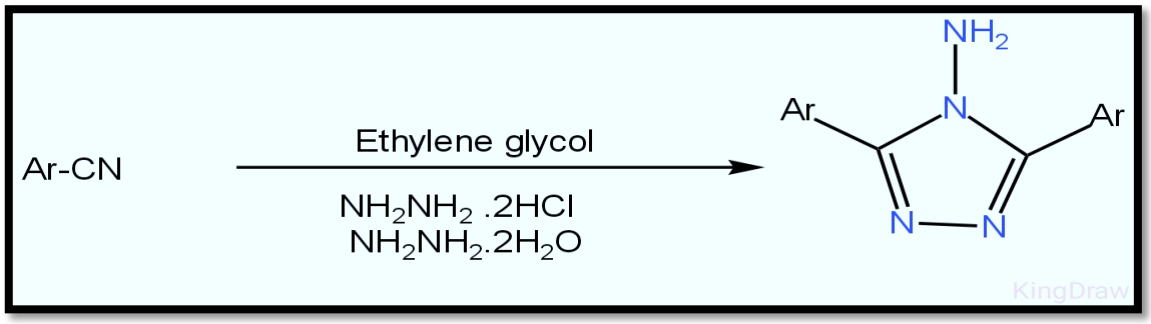

Fig 4.

New antifungal azoles, such as 1,2,4-triazole derivatives, were synthesized by Kidwai et al. 21 employing microwave irradiation and a variety of solid supports **(Fig 5.)**.

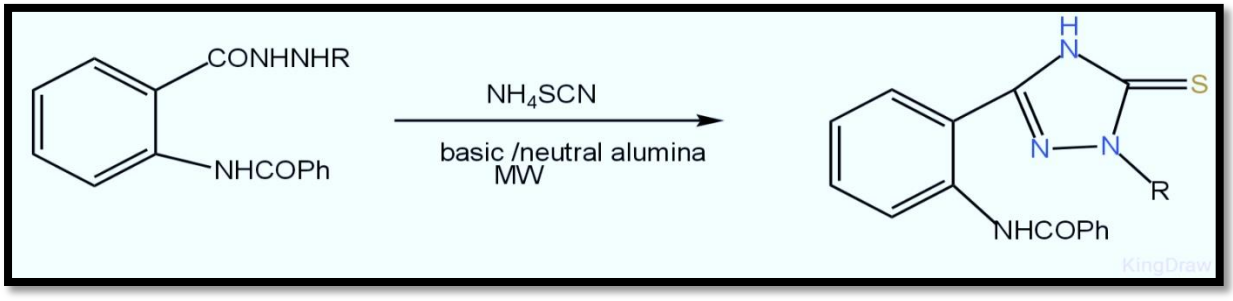

Fig 5.

❖ **TRIAZINES**

Zhijian Zhao et al. applied microwave technology to the synthesis of 1,2,4-triazines, leading to the quick synthesis of several 3,5,6-trisubstituted 1,2,4-triazines in outstanding yield and purity, including many hitherto unrecognized 3-heterocyclic-1,2,4-triazines. Heravi et al.

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used a microwave-assisted, solvent-free condensation of thiosemicarbazide with diketons (RCOCOR, R H, Ph, CH3) to produce 1,2,4-triazines (**Fig 6.).**

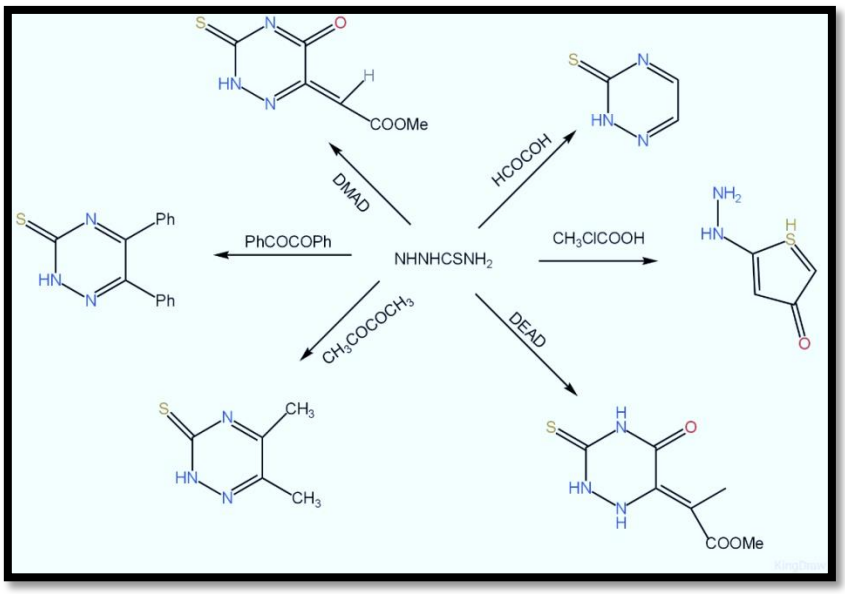

Fig 6.

❖ **BENZIMIDAZOLES AND IMIDAZOLES**

Using microwave and fluorous technologies, the Ugi/de-Boc/cyclization technique for building heterocyclic molecules has been made more efficient. Ugi reactions are used to synthesize substituted quinoxalinones and benzimidazoles using a fluorous-Boc protected diamine. Solid-phase extraction (SPE) over FluoroFlash cartridges30 is used to purify the reaction mixtures, which undergo fast Ugi and post-condensation reactions when exposed to microwaves **(Fig 7.)**.

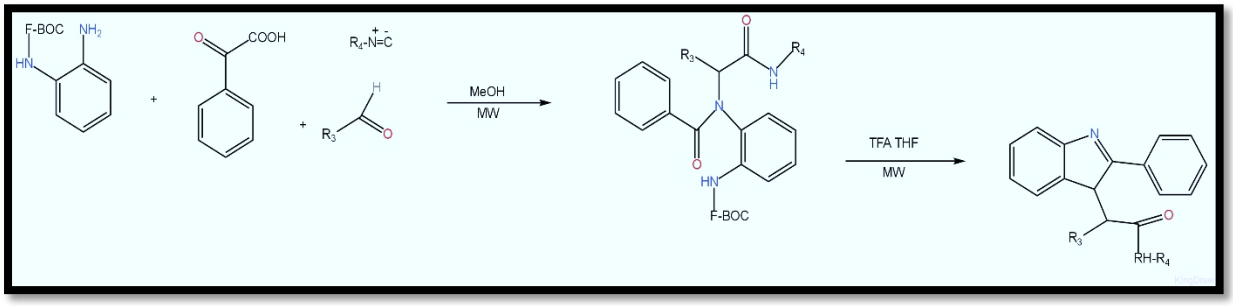

Fig 7.

❖ **BENZOXAZOLES**

In a typical two-step process, a base catalyzed bisacylation of ortho-aminophenols is followed by a Lewis acid-assisted cyclization-dehydration reaction, yielding a benzoxazole with a molecular weight (MW) below 100. Without the need for a base or Lewis acid49,

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benzoxazoles could be produced in a single pot by microwaving acid chlorides and ortho-aminophenols in sealed reaction vessels (**Fig 8.**) [19].

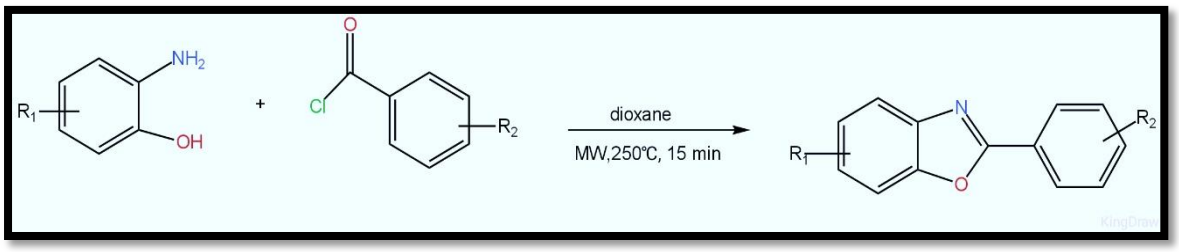

Fig 8.

❖ **1,2,4 OXADIAZOLES AND 1,3,4-TRIS(OXADIAZOLYL)**

The 4-[3-(aryl)-1,2,4-oxadiazol-5-yl]-butan-2-ones were synthesized by de Freitas et al. using methyl levulinate and arylamidoximes. No solvent was used, and the reaction was completed in a fraction of the time and with yields on par with traditional heating methods in a microwave oven.

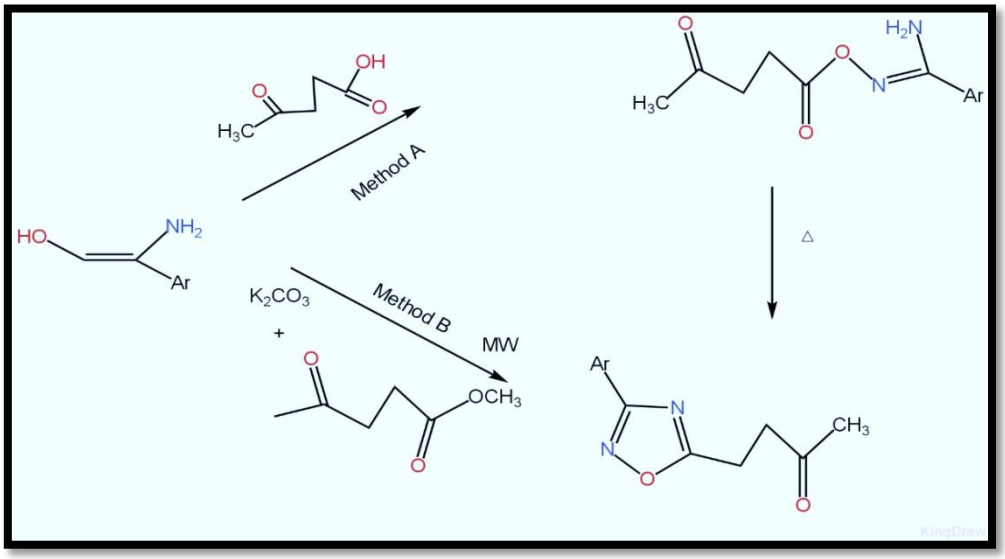

Fig 9.

❖ **BENZOTHIAZOLES**

Preparative methods where the fused thiazole ring is generated from acyclic reactants determine the accessibility of 2-substituted benzothiazoles. In this diagram, we see the many synthetic approaches that could be taken to build a benzothiazoles moiety.

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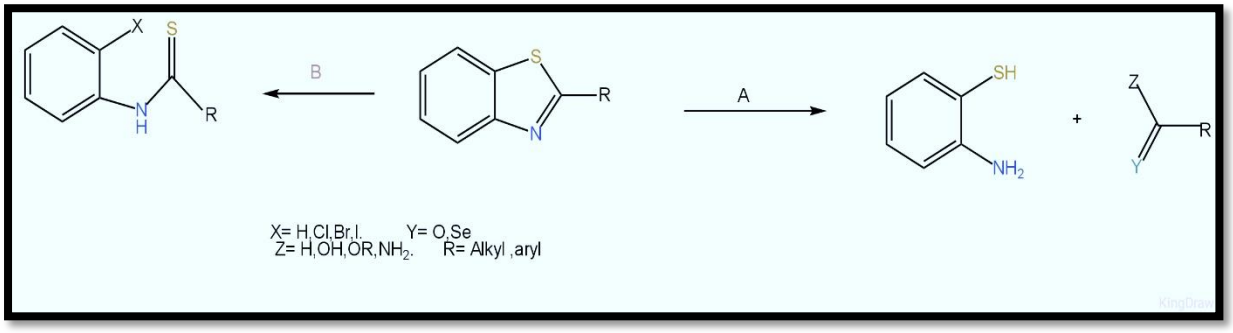

Fig 10.

Condensation of a dinucleophile, such as 2-aminothiophenol, with an ortho-ester in the presence of KSF clay in a monomode microwave reactor running at 60W in a nitrogen atmosphere is one method for the microwave-assisted synthesis of benzothiazoles that has been published.

Fig 11.

In addition, the microwave-assisted, solvent-free synthesis of benzothiazoles via dinucleophile attack on benzaldehydes and benzaldoximines was described.

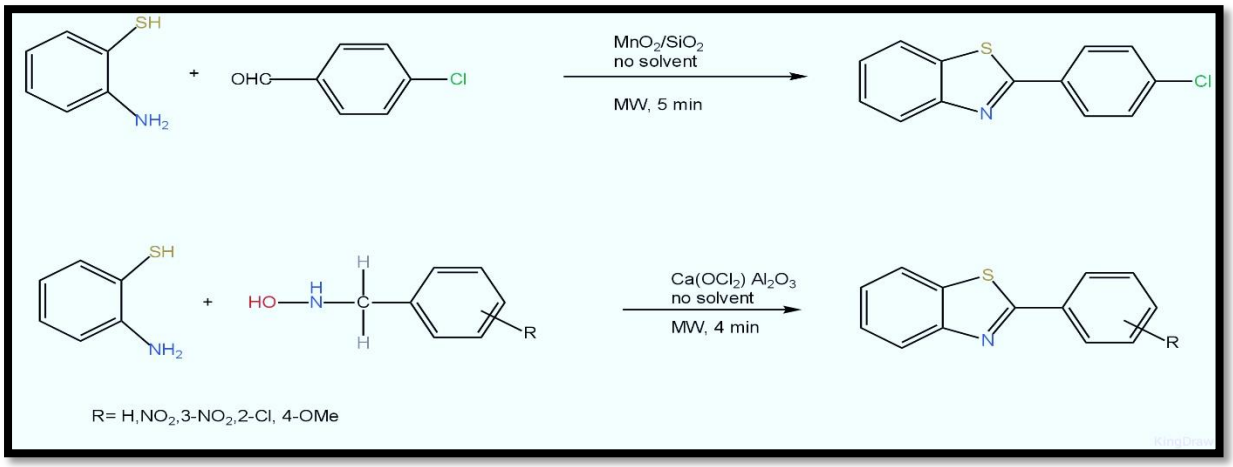

Fig 12.

In a multimode microwave oven, 2-(1,1,1-trifluoroacetonyl) benzothiazole ring was obtained by condensing trifluoroacetyl ketene diethyl acetal with 2- aminothiophenol in the presence of toluene.

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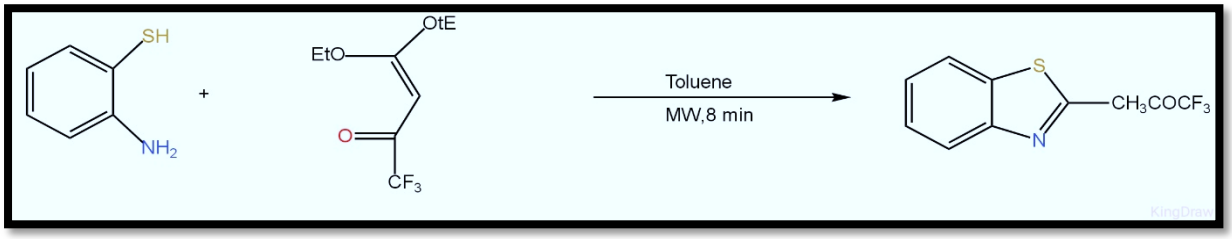

Fig 13.

Oxadiazole derivatives with their therapeutic potentials

Oxolamine, which has a 1,2,4-oxadiazole ring, is used as a cough suppressor; similarly, prenoxdiazine is also used to cure coughing. These and other medicinally active drug molecules having oxadiazole moiety are utilized clinically for the treatment of various disease conditions (Figure 3). The IUPAC notation for this compound is 1,2,4-oxadiazole-3-(2,2-diphenylethyl)-5-(2-piperidin-1-ylethyl). It is a medication prescribed for GI dysfunction. N′,N′-dibutyl-N-(3-phenyl-1,2,4-oxadiazol-5-yl)ethane-1,2-diamine is the chemical name for butalamine, a vasodilator [20].

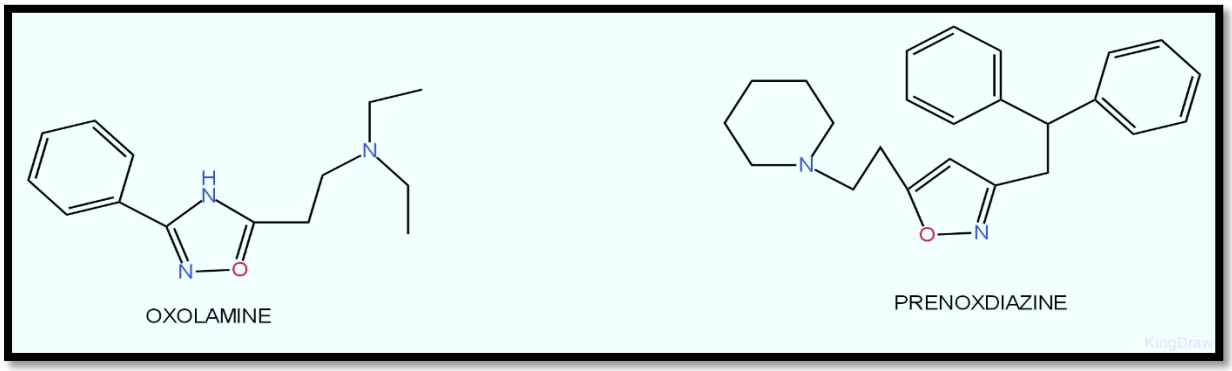

Fig 14.

Oxadiazole derivatives as antioxidant and antimicrobial agent

For example, Farshori et al. reported the simple one-pot synthesis of novel 2,5-disubstituted-1,3,4- oxadiazoles under conventional and microwave conditions (Scheme 8) and evaluated their in vitro antimicrobial activities. The antibacterial efficacy of the newly produced compounds is tested in vitro **(Fig 15.)**.

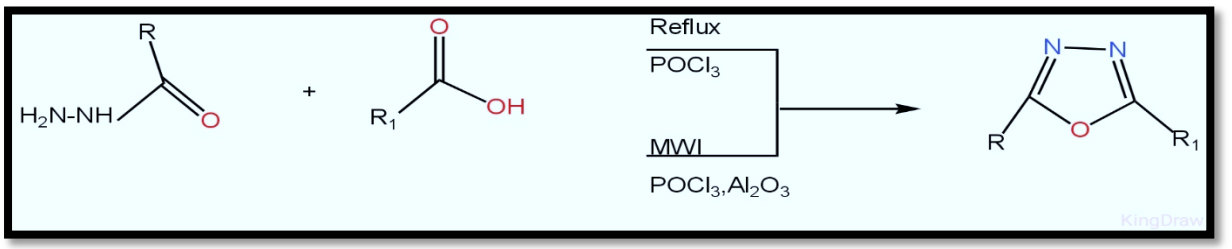

Fig 15.

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Biological effects of oxadiazole analogues

Purity and reproducibility of milligram-to-gram-scale tests without changing reaction conditions for effective use of oxadiazole derivatives as analgesics and anti-inflammatory agents. When compared to traditional heating methods, it allows for more exact control over temperature and pressure conditions. Microwave radiation is absorbed directly by the reactant molecules in a solvent-less or solvent-free reaction. Oxadiazole Derivatives as Analgesic and Anti-Inflammatory Agents Hyperalgesia occurs when nociceptors or peripheral nerves increase their sensitivity to pain. Analgesics are pain-relieving drugs that alter nonessential brain processes while selectively affecting central nervous system and peripheral pain mediators. Both narcotic and non-narcotic analgesics are available. When the body is exposed to infectious pathogens or to physical or chemical harm, it triggers a complicated biochemical response known as inflammation. There are three distinct types of inflammation: the short-term (a few days), the intermediate (two to six weeks), and the long-term (months or years). A message that stimulates inflammatory reactions by acting on blood vessels or cells is called an inflammatory mediator. Prostaglandins (PGs), inflammatory cytokines like IL-1, TNF-, IL-6, and IL-15, and chemokines like IL-8 and GRO-alpha are all examples of inflammatory mediators. The degree and source of inflammation are key factors in determining treatment. It is necessary to create new medications for the treatment of pain and inflammation without the frequent adverse effects of NSAIDs (gas, bloating, heartburn, ulcer, stomach discomfort).

CONCLUSION

As a result, the oxadiazole derivatives are more effective than nonsteroidal anti-inflammatory drugs (NSAIDs) in treating pain and inflammation. In step-I, a mixture of isoniazid e cause and severity was used in the microwave-assisted synthesis reported by Bijuet al. for analgesic and anti-inflammatory activity. It is necessary to create new medications for the treatment of pain and inflammation without the frequent adverse effects of NSAIDs (gas, bloating, heartburn, ulcer, stomach discomfort).

In conclusion, the oxadiazole derivatives are more effective than nonsteroidal anti-inflammatory drugs (NSAIDs) in treating pain and inflammation-induced arthritis. The compounds of 1,3,4-oxadiazole were synthesized using microwave assistance technique.

FUNDING

Nil.

CONFLICT OF INTEREST

None.

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