



EXPEDITIOUS SYNTHESIS OF OXYGEN AND SULFUR HETEROCYCLES BY MICROWAVE

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Introduction

The oxygen- and sulfur-heterocycles are crucial compounds due to their medicinal activities. Microwave-mediated preparation of O-heterocycles and S-heterocycles is an important objective. Many of these reactions are environmentally benign, fast, high yielding and economical. For simplicity, no structures of the compounds are given in this perspective. All references for the synthesis of oxygen and sulfur-heterocyclic compounds are mentioned. In continuation of our diverse research program, we have identified numerous methods for publications in journals and books. We believe readers will find the strategy useful as described herein in their research program. This type of style may open up a new possibility of publishing papers in a concise way.

Oxygen-containing molecules

A few oxygen-containing molecules are mentioned here. Xanthenes are important compounds [1]. For example, benzoxanthenes have anticancer [2], antibacterial [3], and glycoprotein inhibiting properties [4]. Xanthenes are also used in laser methods [5], fluorescent materials [6] and dyes [7].

Ashok et al. described a route for the synthesis of pyranoxanthenes using a one-pot multi-component reaction [8]. The reaction of aromatic aldehyde, substituted chroman-7-ol, and cyclohexanedione was conducted in a microwave in the presence acetic acid. It was shown that microwave heating was the best choice for this purpose. These compounds were tested for antimicrobial and antioxidant properties. A few molecules demonstrated good antibacterial properties. The nitro compound showed good antibacterial activity.

An efficient method for the synthesis of octahydroxanthene by condensation of dimethyl-cyclohexanedione and aldehyde in the presence of an ionic liquid using microwave was conducted [9].

Chavez et al. studied microwave-mediated bismuth triiodide-mediated synthesis of octahydroxanthenes [10]. Microwave-assisted reaction of cyclohexanedione with aldehyde in the presence of bismuth salts was performed. Other bismuth (III) salts were tested. Bismuth nitrate, bismuth halides, bismuth oxide, and bismuth subnitrate were used. Bismuth iodide produced the best yields.

Furans have numerous medicinal properties: anticancer, antibiotics, antifungal, antidepressant, and pain killing. Ashok et al. synthesized a few chroman-4-one fused benzofurans through a cyclization method in a microwave [11]. Chalcone on ring cyclization with butanone produced benzofuran. These molecules were tested to find their *in vitro* antibacterial activity against two Gram-positive bacteria (*Staphylococcus aureus* and *Bacillus faecalis*) and two Gram-negative strains (*Escherichia coli* and *Klebsiella pneumonia*). Two compounds showed antibacterial properties. These compounds were tested for antifungal activity against pathogenic fungi (*Fusarium oxysporum* and *Aspergillus flavus*) and the results are impressive.

Microwave-mediated procedure was employed for a one-pot method for the synthesis of isobenzofuran-ones with sulphamic acid [12]. Isobenzofuran-one was synthesized using 2-carboxybenzaldehyde and many ketones. A reaction of 2-carboxybenzaldehyde, ketone and sulphamic acid on irradiation by microwave produced the pure compound.

The microwave-induced one-pot procedure between furan and furanone were conducted by triethylamine-mediated cascade reaction of furan, benzofuran carboxylic acids and cyanopropargylic alcohols [13]. The product furanone was obtained. The reaction failed to proceed without Et₃N or other tertiary amines.

An aurone has a benzofuran connected to a benzylidene system in position 2. Aurones have many medicinal activities as antioxidant [14], antitumor [15, 16], antiparasitic [17, 18] antiviral, antidiabetic [19], neuroprotective [20], CDK1 inhibitor [21], antinociceptive [22], antimutagenic [23], antioxidant [24], and antidiabetic [25, 26]. The microwave-induced method for the preparation of 2-arylidene-2H-furo[2,3-f]chromen-3(7H)-ones was reported [27]. Microwave-induced Claisen–Schmidt reaction of 1-(5-hydroxy-2H-chromen-6-yl)ethanone and aromatic aldehydes in presence of KOH gave 3-(aryl)-1-(5-hydroxy-2H-chromen-6-yl)prop-2-en-1-one. These molecules were screened for antioxidant and antimicrobial activity. The results showed a high radical scavenging potential and better antimicrobial activity than the available compounds in this area.

The preparation of benzofuran-3(2H)-ones using microwave was investigated by Hu et al. [28]. The products were obtained in satisfactory yields.

5-Ethoxymethylfurfural (EMF) was synthesized from D-fructose in the presence of eutectic solvents (DESs) as promoters in a microwave oven [29]. The choline chloride-oxalic acid produced the best yield of D-fructose. The reaction of D-fructose with ethanol in the presence of DESs gave to 5-Hydroxymethyl furfural (HMF) which on reaction produced 5-Ethoxymethylfurfural (EMF).

Coumarin, an important type of organic molecule was first realized by Vogel in 1820 [30, 31]. In 1868, Perkin prepared coumarin [32]. Many coumarins are medically active [33-38]. Microwave-assisted method was used for the synthesis of cinnamic acid and coumarin [39]. The preparation started with an aldehyde as a substrate and a Wittig reagent. A reaction between aldehyde and ylide was conducted to prepare cinnamic acid derivatives. Microwave-induced synthesis of 3-aryl-furo[3,2-c]coumarins was accomplished by two methods [40]. Numerous 3-aryl-furo[3,2-c]coumarins were synthesized reacting 4-hydroxy coumarins with 2-aryl-1-nitro ethenes. A number of 3-aryl-furo[3,2-c]coumarins were synthesized reacting 4-hydroxy coumarins with aroylmethyl bromides following Feist–Benary's method.

These molecules were screened against Gram-negative bacteria (*Escherichia coli*), Gram-positive bacteria (*Bacillus subtilis*) and fungi *Candida albican*. The agar cup diffusion method was chosen for the determination of the antimicrobial activity. These molecules have good activity against Gram-negative bacteria. Importantly, not a single compound from this group showed superior activity against *Bacillus subtilis* and *Candida albicans*.

Osthole, a natural coumarin has antiarrhythmia, antifungal, antidiabetic, antitumor, antiinflammatory, antiosteoporosis, hepatoprotection, and neuroprotection activities [41-49]. Microwave-assisted synthesis of ostholes by the reaction of 4-hydroxycoumarins and beta-ketoesters was available [50]. These compounds were screened against six phytopathogenic fungi and a few of them showed activity. Many condensed coumarin analogues, pyrano[3,2-c]chromene-2,5-diones were synthesized following microwave-induced procedure [51]. These molecules were screened for their antifungal activity. A few of the coumarin derivatives have antifungal activities.

Numerous hydroxycoumarins were synthesized by microwave-assisted process [52]. On reaction of coumarins with chloroacetonitrile and potassium carbonate resulted in O-substituted cyanomethoxy derivatives. The molecules having coumarin and chromene systems, called pyranocoumarins are challenging and pyranocoumarins demonstrated anti-HIV activity.

Ashok et al. showed the synthesis of this type of compound using microwave-mediated process [53]. These compounds were screened for their antimicrobial activity. Some of them was active against fungal and bacterial strains.

Microwave-assisted palladium-induced Heck reaction was conducted on quercetin derivative with terminal alkenes [54]. Synthesis of substituted 1,4-dioxines via oxiranes and diazo compounds was known. These molecules exhibited urease inhibition and cytotoxic activity.

Sulfur containing molecules are biologically crucial [55-57]. Microwave-assisted parallel synthesis of a library of N-aryl and N-aryl thioureas were performed [58].

Microwave-mediated copper-assisted ring expansions of heterocycles with diazo-dicarbonyl molecules were studied [59]. This method described a simple procedure for the preparation of dioxins. Preparation of oxathiines by a ring enlargement of thiranes and diazo compounds was described.

A few thiophene hydrazones were synthesized by microwave-induced method [60]. A few chemists prepared sulfanyl-substituted chromenones. Anjaiah et al. demonstrated microwave-assisted synthesis of 3-arylsulfanyl-chromen-2-ones by condensation of arenesulfonohydrazides with hydroxy-chromenone using iodine [61]. These molecules were tested for *in vitro* antimicrobial activity and good results were observed.

Various 1-(arylthio)naphthalen-2-ols were synthesized by condensation of naphthalen-2-ol with diaryldisulfanes using iodine and microwave [62]. These compounds were tested to identify their antibacterial and antifungal properties.

A microwave-induced method for the preparation of hetaryl and ferrocenyl substituted thioketones was described [63]. Microwave-mediated one-pot preparation of oxathiolanes was conducted by Kermani [64]. The 2-(nitromethylidene)-1,3-oxathiolane was prepared using nitromethane, CS₂, oxirane, and Et₃N. Some substituted hetaryl thioketones reacted with diazoketones in a microwave [65]. The phenyl(thiophen-2-yl) thioketone on reaction with 2-diazo-1,2-diphenylethanone in the presence of microwave produced 2,4,5-triphenyl-2-(thiophen-2-yl)-1,3-oxathiole and 3,3,4-triphenyl-4-(thiophen-2-yl)thietan-2-one. Similar studies with ferrocenyl compounds under microwave irradiation were not successful. But, these reacted with diazopropanone and 2-diazo-ethanone with LiClO₄ to give α,β -unsaturated ketones.

Benzoselenophene moiety is regarded as a bioisoster of benzofuran, naphthalene, benzothiophene, and indole system [66]. Benzoselenophenes have received attention because of their uses in medicines [67-70]. Arsenyan et al. reported microwave-induced palladium-promoted cyanation of 3-bromo-2-(1-hydroxyalkyl)benzo[b]selenophenes [71]. The starting molecule was activated in a microwave irradiation and catalyst Pd(PPh₃)₄.

Various phosphinic, phosphonic esters, and phosphine oxides were synthesized by P-C-mediated reactions of aryl- and vinyl halides with phosphites, phosphinates and phosphine oxides using microwave [73]. Microwave-induced method was applied for the synthesis of arylphosphonates using cyclodiphosphazane-Pd [74]. Keglevich et al. demonstrated that Hirao reaction proceeds with excess P-reagents [75]. Jablonkai and Keglevich showed a ligand-free Carbon-Phosphorous coupling method [76]. A reaction of halo benzoic acids (iodo and bromo), and diaryl phosphine oxides was conducted in a microwave. Microwave-assisted preparation of azidoalkylphosphonates through nucleophilic reaction of the bromoalkylphosphonates with sodium azide was reported [77].

An isomerization of 7-substituted cycloheptatrienes was performed with DABCO and t-BuOK [78]. Following this method, allyltriphenylphosphonium bromide was prepared by reaction of triphenylphosphine and allyl bromide.

The AuNP-supported Gd complex was an effective promoter for the acetylation of alcohols and phenol [79]. Microwave-assisted acetylation of alcohols and phenol was performed. Microwave-assisted Cannizzaro reaction was reported to convert aldehydes to carboxylic acids [80]. A microwave-mediated Claisen-Schmidt method for the synthesis of α,β -unsaturated ketones was known [81]. The preparation of 2,3-dihydroxypropyl decanoate was accomplished by esterification of decanoic acid with glycidol and glycerol carbonate [82]. Monoglycerides were synthesized from decanoic acid, and glycerol carbonate or glycidol in the presence of TBAI.

The α -aryl malonates are important for the synthesis of heterocycles [83]. The reaction of aromatic halides with diethyl malonate using $\text{Cu}(\text{OTf})_2$, Cs_2CO_3 , and 2-picolinic acid was performed for this purpose.

Conclusion

The significance of microwave-mediated reactions for the synthesis of numerous organic compounds is discussed here. Many microwave-induced processes toward oxygen and sulfur molecules are described. These methods have numerous advantages: solvent-free reaction, reduction of reaction time, catalytic nature, economical and rapid process. Because of these advantages, researchers engaged in drug discovery use microwave-induced procedures.

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