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Recent Advances in the Synthesis of Benzothiazoles : A Review

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ABSTRACT

Benzothiazoles are important heterocyclic compound because of their wide range of biological and pharmacological activity. In literature, it is reported that Benzothiazoles and its derivatives possess numerous pharmacological activities like antitumor, anticonvulsant, antimicrobial, antioxidant, antitubercular, antimalarial, antifungal, anti-inflammatory and antidiabetic activities. In recent year several methods have been developed for the synthesis of Benzothiazole derivatives due to their importance in bioorganic and medicinal chemistry. The present review focuses on systematic literature report of various synthetic procedures of Benzothiazoles derivatives using different strategies from the period 2010 to 2021.

Keywords: Benzothiazole, Pharmacological activities, Synthesis.

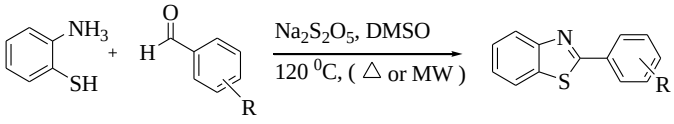
I. INTRODUCTION

Heterocyclic chemistry is an important branch of organic chemistry. Heterocyclic compounds are cyclic compounds with the ring containing carbon and other element such as oxygen, nitrogen and sulphur. Benzothiazole is a sulphur containing heterocyclic compound which contain benzene ring fused to thiazole ring. Benzothiazole and their derivatives are biologically important scaffold owing to their potent antitumor [1], anticancer [2], anti-bacterial [3], antiviral [4], Anthelmintic [5], Anti-diabetic [6], Anti-trypanosomal [7], Anti-oxidant [8], Anti-glutamate and Anti-parkinsonism [9, 10], Aanalgesic [11], Anticonvulsant [12], Neuroprotective [13], Vasodilator [14], Muscle relaxant activities [15]. In industry, they are also used in the production of antioxidant [16], vulcanization accelerators [17] and organic optoelectronic materials [18]. The 2-arylbenzothiazoles possess utility as imaging agents for β-amyloid, antituberculotic, chemiluminescent agents, calcium channel antagonists, antiparasitics, and photosensitizers [19].

In recent years, because of their importance in bioorganic and medicinal chemistry, several methods have been developed for synthesis of benzothiazole and its derivatives. Therefore, the present review mainly focuses on the research work reported in the recent scientific literature in synthesis of benzothiazole derivatives.

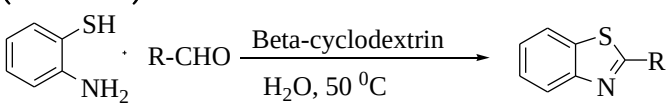
II. SYNTHESIS OF BENZOTHIAZOLE

A. A. Weekes et al [20] have reported simple one-step method for the synthesis of 2-phenylbenzothiazoles using sodium metabisulfite as an oxidant by condensation of 2-aminothiophenol and substituted benzaldehyde. The reaction was carried out in both thermal and microwave condition using DMSO as solvent (Scheme-1). Further to explore the scope of the reaction both electron-withdrawing and electron-donating groups on the benzaldehyde were examined. Both the aldehyde having electron donating and withdrawing group gives excellent yield of product with simple workup and isolation. The advantages of this protocol are excellent yields under both microwave-assisted or thermal conditions and simple product isolation without the need for chromatographic purification.



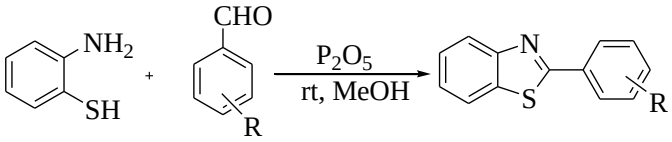
Scheme-1

B. S. Londhe et al [21] have described the synthesis of 2-arylbenzothizole by the condensation of 2-aminothiaophenol and aryl/heteryl aldehyde using biomimetic catalyst β-cyclodextrin in water. Further, the reaction was carried out by changing the concentration of β- cyclodextrin and reaction temperature. It was observed that when equimolar quantities of the alcoholic concentrated solution of aldehyde and 2-aminothiophenol were stirred in equimolar aqueous (5.6 %) solution of β-Cyclodextrin at 50 °C better yield of benzothiazole were obtained. This protocol has many advantages such as low temperature, neutral pH, atmospheric pressure, absence of acidic/ metallic catalyst and readily available catalyst which can be recycled and reusable **(Scheme-2)**.



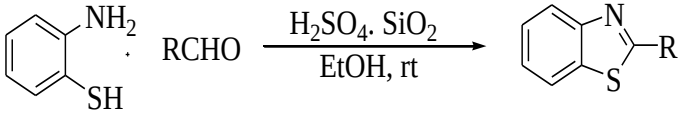
Scheme-2

S. V. Bhosale and Co-worker [22] have reported the synthesis of 2-arylbenzothiazole by the condensation of 2-aminothiophenol with aromatic aldehyde using P_2O_5 catalyst in methanol as solvent at ambient temperature. The reagent phosphorous pentaoxide used in this reaction is less expensive acid catalyst which is water soluble and easily removed. Different aldehydes with various substituents were exposed to the reaction condition and it was found the yield of benzaldehyde is more compare to the substituted aldehyde. Furthermore, heteroaromatic and aliphatic aldehyde were employed for the synthesis of 2-substituted benzothiazole. Good yield were obtained with heteroaromatic aldehyde and low yield of product with aliphatic aldehyde. This method offers several advantages such as inexpensive catalyst, short reaction times, cleaner reactions and good yields of products. **(Scheme-3)**



Scheme-3

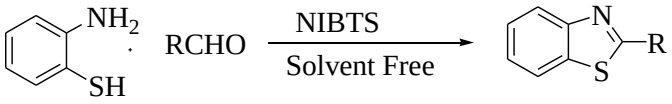
2-arylbenzothiazole obtained from the condensation reaction of aldehyde with 2-aminothiophenol using sulphuric acid Immobilized on silica as reusable catalyst under heterogeneous and mild condition in ethanol at room temperature gives high yield which was pointed out by Behrooz Maleki and Co-author **(Scheme-4)** [23].They have shown that $H_2SO_4.SiO_2$ is a cheap, reusable and eco-friendly catalyst for the synthesis of 2-arylbenzothiazole.



Scheme-4

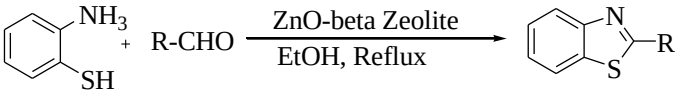
V. Hojat et al [24] found that *N*, *N*'-Diiodo-*N*, *N*'-1, 2-ethandiylbis (*p*-toluene sulfonamide) (NIBTS) is a good and new reagent for synthesis of 2-arylbenzothiazoles by the reaction of 2-aminothiophenol and aldehyde at room temperature

under solvent-free condition with good to high yield. In this protocol excellent yield of 2-arylbenzothiazole were obtained with aromatic aldehyde having electron donating and withdrawing group but only 30% yield of product were obtained with Octanal an aliphatic aldehyde. The method has advantages in terms of product yields, absence of solvent, short reaction times, non-corrosive and easy work up of reactions **(Scheme-5).**



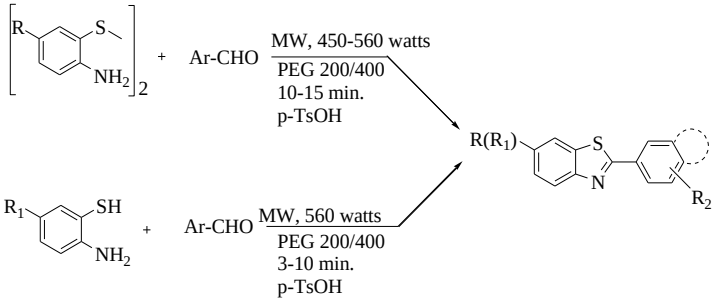
Scheme-5

M. K. Lande and Co-worker [25] used ZnO-beta Zeolite as an effective and eco-friendly heterogeneous catalyst for the synthesis of benzothiazole derivatives using of 2-aminothiophenol and aldehyde in ethanol under reflux condition. Initially, the catalyst ZnO-beta Zeolite was prepared and characterized by FT-IR, XRD, SEM, EDS, TPD and BET analysis. Excellent yield of product were obtained with aldehyde bearing electron donating and withdrawing group. High yields, simple experimental procedure and reusability of the catalyst are main advantages of this procedure. **(Scheme-6)**



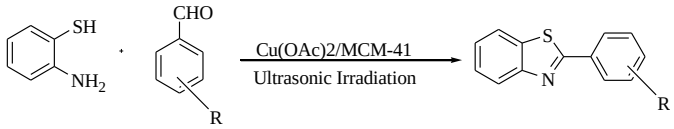
Scheme-6

T. G. Deligeorgiev and co-authors [26] have successfully developed green procedure for the synthesis of 2-aryl- and 2-hetarylbenzothiazoles by condensation of 2, 2-diaminodiphenyldisulfides or 2-aminothiophenols and aromatic aldehydes in PEG 200/400 under microwave irradiation. The advantages of this procedure are short reaction time, easy workup procedure, highly reproducible, use of green solvent, high yield and high purity of product. **(Scheme-7)**



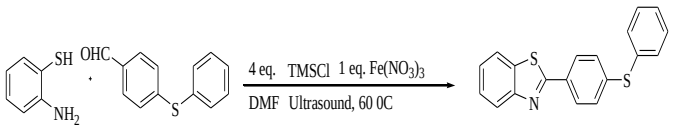
Scheme-7

S. Sadjadi and H. Sepehrian [27] have developed a new convenient method for the synthesis of 2-arylbenzothiazole using o-aminothiophenol and aromatic aldehyde in the presence of Cu $(OAc)_2$/MCM-41 catalyst under ultrasonic irradiation. Firstly, the catalyst was prepared and characterized by XRD and BET analysis. Excellent yield of desired product were obtained with aldehyde having electron donating and withdrawing group. The main advantages of this method are short reaction time, easy and quick isolation of product, reusability of catalyst and excellent yield. **(Scheme-8)**



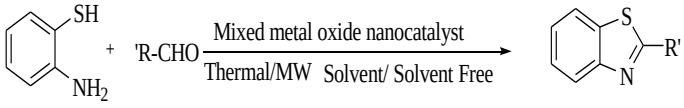
Scheme-8

Y. Yuan and S. Guo [28] have reported a convenient one-pot cyclocondensation method for synthesis of benzothiazole from aromatic ortho-aminothiophenol and aldehydes using chlorotrimethylsilane in dimethylformamide as promoter and water scavenger under ultrasonic irradiation. The best result of this protocol was obtained with 2 aminothiophenol, aldehyde, TMSCl and Fe $(NO_3)_3$ (1:1:4:1) at 60 °C for 30 min. under ultrasonic irradiation in DMF or without solvent. Low cost, ready availability of reagents and simple procedures for conducting the reaction and isolating the products are the advantageous of this method. **(Scheme-9)**



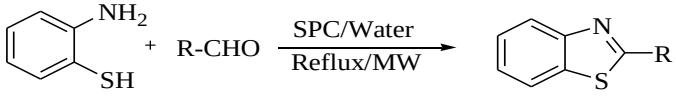
Scheme-9

P. Bandyopadhyay and Co-worker [29] have described mesoporous mixed metal oxide nanocrystals of Al_2O_3–Fe_2O_3, Al_2O_3–V_2O_5 and Al_2O_3–CuO as heterogeneous catalysts for the synthesis of 2-substituted benzothiazoles using 2-aminothiophenol and aromatic aldehyde. These nanocrystalline catalysts showed significant catalytic activity with a high substrate to catalyst weight ratio (20:1) with 81–96% yield. In comparison to conventional heating the solvent-free microwave assisted synthesis of these compounds that gives excellent yields in much lesser time (0.75–1.5 min). This one-pot, mixed metal oxide catalyzed synthetic method is an unprecedented, inexpensive and rapid alternative for the preparation of benzothiazole derivatives. This method has several advantageous such as greater selectivity, cost-efficiency, clean reaction profiles, simple work-up procedure and high yields. **(Scheme-10)**



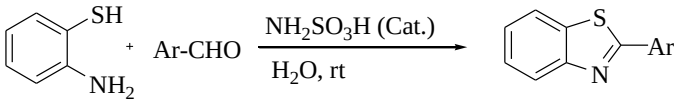
Scheme-10

A. Shokrolahi et al [30] have reported Sulfonated porous carbon (SPC) as heterogeneous catalyst for the synthesis of benzothiazole derivatives in water by the condensation of 2-aminothiophenol and aldehydes. In this protocol various aromatic and heteroaromatic aldehyde were used for the synthesis of 2-aryl benzothiazole. It was found that under optimal condition the electron donating and withdrawing substituent on aromatic aldehyde have no impact on the yield and reaction time. The remarkable advantageous of this methodology are mild reaction conditions, clean reaction profiles, small quantity of catalyst and simple workup procedure. **(Scheme-11)**



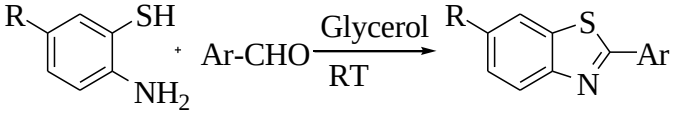
Scheme-11

An efficient protocol was developed by A. Rostami and A. Yari [31] for the synthesis of 2-Arylbenzothiazoles from condensation of 2-aminothiophenol and aromatic aldehydes using catalytic amount of sulfamic acid at room temperature. Further the reaction was carried out using structurally diverse aromatic aldehyde such as substituted benzaldehyde, 1-naphthaldehyde, 2-naphthaldehyde, 9-anthraldehyde and terephthaldehyde. It was found that under optimal condition the substitution on aromatic ring (donating or withdrawing) have no effects on the yield and reaction time. The use of the eco-friendly, cost-effective, commercially available, reusable and chemically stable catalyst, no use of any additional oxidants, short reaction time, mild reaction conditions and very easy work-up, avoiding hazardous and toxic solvent, good to high yields of products are the important advantageous of this method. **(Scheme-12)**



Scheme-12

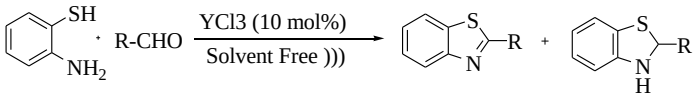
Green and highly efficient strategy has been developed for the synthesis of 2-arylbenzothiazoles from the reaction of 2-aminothiophenols and aromatic aldehydes without catalyst in glycerol as a green solvent at ambient temperature by K. U. Sadek and Co-worker [32]. The rate of reaction and yield of the product slightly depend upon substituent on aromatic aldehyde. The yield of product slightly increased with aldehyde having electron withdrawing group. This methodology has the advantages of being green and highly efficient, simple procedure, easy isolation of product, excellent yield and catalyst free condition at ambient temperature. **(Scheme-13)**



Scheme-13

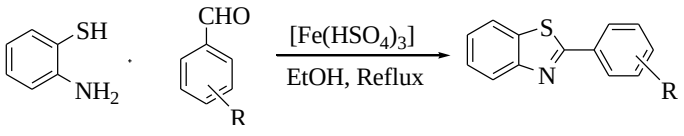
L. Zhang et al [33] have reported simple and green method for the synthesis of 2-substituted benzothiazoles using rare-earth metal chlorides as efficient catalysts under ultrasound irradiation. In this reactions the products of 2-substituted benzothiazoles

were obtained in excellent yields using a stoichiometric amount of aromatic or aliphatic aldehyde with 2-aminothiophenol (1:1) catalyzed by 10 mol% YCl_3 for 8 h under ultrasonic irradiation. It was observed that reactions of aliphatic aldehydes with 2-aminothiophenol resulted in 2, 3-dihydrobenzothiazoles derivatives instead of the products of 2-substituted benzothiazoles. The advantages of present method are broad generality, economical catalysts, good yields, solvent-free condition and simple operation. **(Scheme-14)**



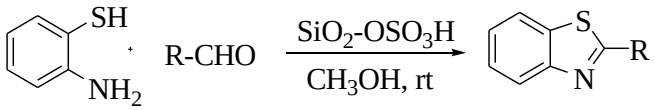
Scheme-14

A series of substituted benzothiazoles were prepared by H. Eshghi and Co-author [34] from one-pot reaction of o-aminothiophenol with various aldehydes in the presence of ferric hydrogensulfate both in EtOH and water as solvent. Furthermore, a variety of aromatic aldehydes including electron withdrawing and electron-donating groups were investigated and do not observe any significant substitution effect according to reaction times. Results indicate that the catalyst plays an important role in this transformation. In the mechanism of the reaction the bifunctional FHS catalyst, having Lewis acidic ferric cation and Bronsted acidic hydrogensulfate function, mediates the carbonyl condensation. Lastly, by an oxidative dehydrogenation gives benzothiazole compounds. This process has several advantages like short reaction time, easy and quick work-up and excellent chemoselectivity. **(Scheme-15)**



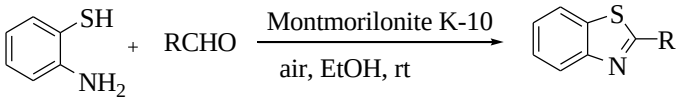
Scheme-15

G. F. Chen and Co-authors [35] have synthesized 2-substituted benzothiazoles via one-pot reaction from aromatic aldehydes and o-aminothiophenol catalyzed by silica sulfuric acid in absolute methanol at room temperature. Further, the effect of temperature and solvents was studied in the presence of silica sulfuric acid using various solvents including EtOH, $CH_3COOC_2H_5$, CH_2Cl_2, CH_3CN, THF, $CHCl_3$, and CH_3OH at room or reflux temperature. The results showed that the product yield was significantly influenced by the solvents and temperature. In general, low yield were obtained in non-polar solvents such as ethyl acetate either at room or reflux temperature. In CH_3OH the best conversion was observed at room temperature. This method is an environmentally friendly and reusable catalyst, a simple procedure, mild conditions, short reaction times, and good to excellent yields of products are the significant advantages of this procedure. **(Scheme-16)**



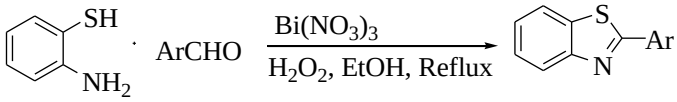
Scheme-16

G. F. Chen et al [36] described simple and eco-friendly synthesis of 2-substituted benzothiazoles from aldehydes and o-aminothiophenol catalyzed by montmorillonite K-10 catalyst with continuous bubbling of air as oxidant in absolute ethanol at room temperature. Further, the reaction was employed with various aldehyde having electron donating and withdrawing group. It was observed that the yield of product depend on the substituent on aromatic aldehyde. The heterocyclic aldehyde such as furfural gives moderate yield whereas aliphatic aldehydes like n-butyraldehyde and n- heptaldehyde were used under same reaction condition none of the desired product was obtained. This method offer several advantages such as simplicity, ease of handling of the system, an environmentally friendly and reusable catalyst, a plentiful, inexpensive, and safe oxidant, and mild reaction conditions. **(Scheme-17)**



Scheme-17

M. Ghashang [37] have developed a simple and eco-friendly procedure for synthesis of 2-arylbenzothiazoles by cyclo-condensation reaction of 2-aminothiophenol and aldehydes in the presence of bismuth nitrate (Bi $(NO_3)_3$) as heterogeneous catalyst in ethanol under reflux with H_2O_2 as oxidant. The optimizations of solvent the author have employed different solvents and solvent-free conditions at higher temperatures. It was observed that the best result was obtained with ethanol as solvent under reflux conditions. Solvent-free conditions gave the product in lower yield and a low-polarity solvent, for example CH_2Cl_2, gives hardly any product. Excellent yields, simple procedure, short reaction times and reusability of catalyst are the advantages of present protocol. **(Scheme-18)**

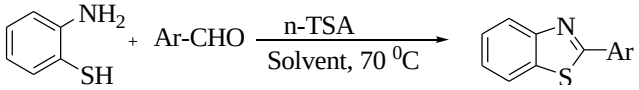


Scheme-18

One-pot synthesis of 2-substituted benzothiazoles from aromatic aldehydes and o-aminothiophenol in the presence of $FeCl_3$/Montmorillonite K-10 in absolute methanol at 25–30 °C under ultrasound irradiation reported by G. F. Chen et al [38] **(Scheme-19)**. An inexpensive and easily available reagent, a simple procedure, mild conditions, short reaction times and moderate to good yields are the remarkable advantages of this procedure.

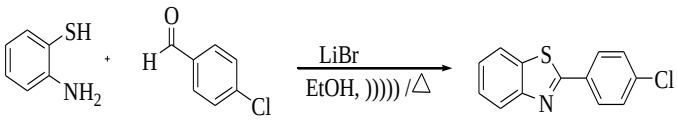
Scheme-19

S. Rahmani et al [39] have used Nano-titania-supported sulfonic acid (n-TSA) as an efficient, inexpensive and reusable heterogeneous nano catalyst for synthesis of 2 arylbenzothiazole derivatives from aldehydes and o-aminothiophenol under solvent free condition **(Scheme-20)**. In this reaction good to excellent yields of product were obtained for different aromatic aldehydes with both electron withdrawing and electron donating groups. This method offer several advantages such as easy preparation and high efficiency of catalyst, operational simplicity, mild reaction conditions, easy purification, high yields of products, no environmental hazards and reusability of the catalyst for at least 7 times.



Scheme-20

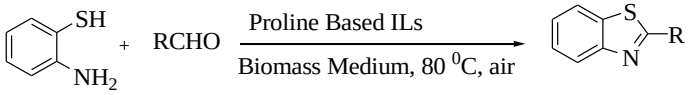
C. H. Gill and Co-workers [40] have developed a new and efficient protocol for synthesis of benzothiazoles from aromatic aldehydes and o-aminothiophenol using lithium bromide as catalyst under environmentally friendly conditions **(Scheme-21)**. Further the reaction was carried out with a variety of electronically divergent aromatic aldehydes with o-aminothiophenol using the conventional and ultrasound irradiation methods. It was observed that the presence of electron-withdrawing and electron-donating groups on the aromatic rings did not affect the product yield. High product yield, utilization of ultrasound irradiation, decreased reaction time and simplified workup procedure are the significant advantages of this synthetic strategy.



Scheme-21

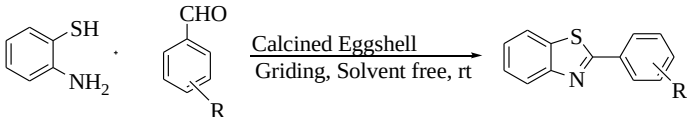
A simple, efficient, and eco-friendly aerobic procedure for synthesis of 2-substituted benzothiazoles were reported by Z. Y. Yu and Co-authors [41] from aldehydes and o-aminothiophenol using proline based ionic liquid as catalyst under air condition. The catalyst tetraethylammonium amino acid ionic liquids were synthesized via simple neutralization reactions. It was observed that excellent yields of the 2-arylbenzothiazoles were obtained with electron-withdrawing substituents possibly because of benzaldehyde with electron-withdrawing substituents were more activated than other substituents. The advantages of this method are

the simple reaction conditions, nontoxic solvents, easy workup and reusability of catalyst. **(Scheme-22)**



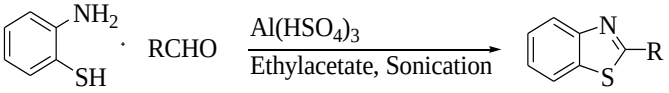
Scheme-22

A. V. Borhade and Co-authors [42] have prepared series of substituted 2-arylbenzothiazoles by reaction of o-aminothiophenol and aromatic aldehydes catalyzed by calcinized eggshell as an efficient and green catalyst under solvent free conditions using a grinding method at room temperature **(Scheme-23)**. The catalyst was prepared from chicken eggshell waste and characterized by different analytical techniques such as FT-IR, XRD, TGA, SEM, and EDAX. Furthermore different aromatic aldehydes were treated with o-aminothiophenol having both electrons withdrawing and electron donating substituents in aromatic aldehyde, it was observed that because of aryl aldehydes with electron-withdrawing substituents are more activated than other substituents gives slight increase in the yield of product. The remarkable advantages of this procedure are the green method, simple work up, excellent yield, solvent-free reactions, and utilization of waste as a reusable catalyst.



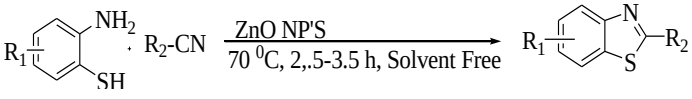
Scheme-23

B. F. Mirjalili and Co-authors [43] have described Al $(HSO_4)_3$ as an efficient catalyst for the synthesis of benzothiazole derivatives by condensation of 2-aminothiophenol and aldehydes under sonication conditions **(Scheme-24)**. Furthermore, the reusability of catalyst was studied and no reusability was observed. In this process the aromatic aldehyde having electron donating and withdrawing group gives high yield and aliphatic aldehyde gives oily liquid which requires difficult method for purification. This method has several advantages such as short reaction time, high yield, a clean process, simple methodology, easy workup and green condition.



Scheme-24

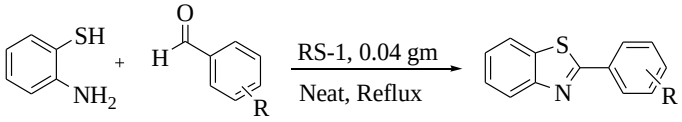
K. D. Dhawale and Co-authors [44] have described the synthesis of 2-substituted benzothiazoles using ZnO-nanoparticles as a catalyst by the condensation of 2-aminothiophenol and aryl/alkyl nitriles under solvent-free reaction conditions **(Scheme-25)**. The ZnO-NPs were prepared by co-precipitation method. The prepared ZnO-NP's were characterized XRD, SEM, TEM and UV-Vis absorption spectrum. The synthesized ZnO-NPs were used as catalyst for the synthesis of 2-substituted benzothiazoles.The reaction was performed in different solvents such as DMF, CH_2Cl_2, THF, DMSO, H_2O, CH_3OH, CH_3CH_2OH and 1,4-dioxane However, excellent yield of product was obtained in ethanol and methanol. But ethanol can be used because it is nontoxic and eco-friendly than methanol. Further this reaction was carried under solvent free condition and excellent yield of product was obtained. The solvent-free reaction has easy product separation and catalyst recycling. Therefore, Author has decided to perform the subsequent reaction under the solvent-free conditions at 70 ^{0}C temperature. The advantages of this method are the faster reaction, functional group compatibility, excellent yield, no column purification, chemo-selectivity, high catalytic activity, recyclability, easy handling, low corrosiveness and environmental compatibility.



Scheme-25

S. P. Gadekar and M. K. Lande [45] have reported Ruthenium silicate (RS-1) zeolite as novel heterogeneous efficient catalyst for synthesis of 2-arylbenzothiazole derivatives from 2-aminothiophenol and substituted aryl aldehyde

(Scheme-26). The catalyst ruthenium silicate (RS-1) and zeolite catalyst have been successfully synthesized by using hydrothermal process. The catalyst was characterized by Fourier transform infrared spectroscopy, scanning electron microscopy, powder X-ray diffraction, Energy dispersive X-ray pattern/spectroscopy analysis. Further, the authors have examined the effect of various solvents such as CH_2Cl_2, EtOH, H_2O, MeCN, DMF and solvent free condition on the reaction rate under the same reaction conditions, solvent free reaction afforded the products in higher yield and shorter reaction time. The amount of catalyst was also optimized. In this procedure it was observed that aromatic aldehydes having electron-withdrawing groups on the aromatic ring react faster than electron-donating groups. Reusability of RS-1 catalyst was investigated and it was observed that, the catalyst can be used for three times runs without loss of the product yield as well as its catalytic activity.



Scheme-26

III. CONCLUSION

In conclusion, this review has summarized recent advances in the synthesis of benzothiazole derivatives. Benzothiazole and its derivative have played an important role in biochemistry, medicinal chemistry and industry. The number of methods for their synthesis has been developed in the last decades. The benzothiazoles were efficiently synthesis by the condensation of 2-aminothiophenol and substituted aryl aldehyde. Here, we have compile recently reported procedure for the synthesis of benzothiazoles. This review will be very advantageous to the researchers who are working in this area of research.

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