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Neat Reaction Strategies for Organic Transformation: A Mini Review

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ABSTRACT

The solvents and reagents used in conventional methods in organic synthesis cause many side effects on human health as well as on the environment. To prevent the side effects, researchers are adopting different ways following the 12 principles of green chemistry. Neat method for organic transformation is the method where reagents and substrates are used without solvents. These reactions typically involve the direct mixing of reactants, often under heat or irradiation, to facilitate chemical transformations. The absence of solvents eliminates waste and reduces the need for solvent disposal, aligning with green chemistry principles. This review outlines the synthesis of different heterocyclic compounds using a neat reaction strategy.

Keywords- ball milling, green chemistry, neat reaction, microwave-assisted and ultrasound-assisted

Introduction

Conventional methods in organic synthesis refer to the methods used to synthesize organic compounds through chemical reactions that include the use of solvents, reagents, catalysts, and heating methods and they require harsh conditions, produce waste, and rely on toxic solvents and reagents. Most commonly used solvents and reagents in synthesis are dichloromethane (DCM), chloroform, acetonitrile, dimethylformamide (DMF), dimethylsulfoxide (DMSO), xylene, benzene and toluene etc.

These conventional methods have adverse side effects on human health as well as on the environment. To prevent these hazards, researchers are adopting different ways in accordance with the 12 principles of green chemistry[1]. Some of them include the grinding or ball milling methods[2], microwave and ultrasound-assisted[3,4], use of green solvents[5,6] and heterogeneous or nanocatalysts[7] and neat(solvent-free) reactions[8,9].

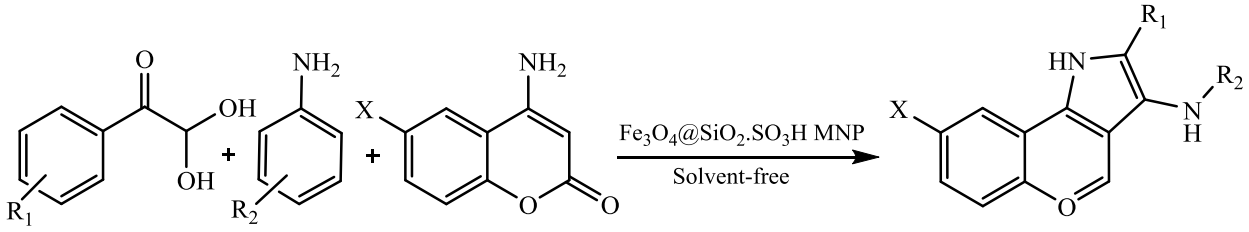
Neat methods for organic transformations are the methods where reagents and substrates are used without solvents. These reactions typically involve the direct mixing of reactants, often under heat or irradiation, to

facilitate chemical transformations. The absence of solvents eliminates waste and reduces the need for solvent disposal, aligning with green chemistry principles. Neat conditions can enhance reaction rates, improve yields, and promote selectivity due to the concentrated nature of the reactants.[10]

Heterocyclic compounds play a very important role as they are frequently found in enzymes, vitamins, and natural products. Most of the drugs contain the heterocyclic nucleus which possesses many biological activities such as anti-inflammatory, antibacterial, antifungal, antioxidant, antiallergic, enzyme inhibitors, anticonvulsant, anti-HIV, antidiabetic, anticancer activity, insecticidal agents, etc.[11]

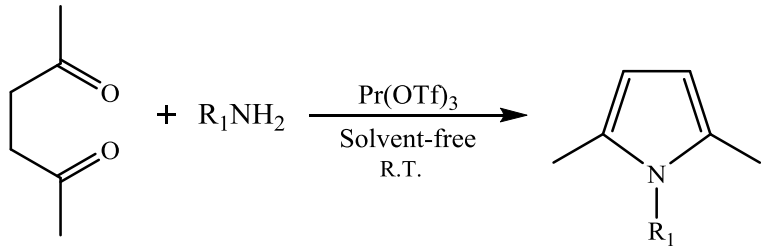
A. NEAT SYNTHESIS OF PYRROLES

Mukherjee et al.[12] demonstrated an environmentally benign one-pot method for the synthesis of chromeno[4,3-b]pyrrol-4(1H)-one derivatives. Three-component domino reaction of 4-aminocoumarins, arylglyoxal monohydrates, and nucleophilic substrates yields functionalized chromeno[4,3-b]pyrrol-4(1H)-ones, catalyzed by $Fe_3O_4@SiO_2$-SO_3H magnetic nanoparticles under solvent-free conditions. The procedure has numerous advantages like atom-economy fashion, short reaction time, good yield of the products, use of a recyclable nanocatalyst, and avoiding the use of hazardous solvents as well as expensive catalysts/reagents. (Scheme-1)



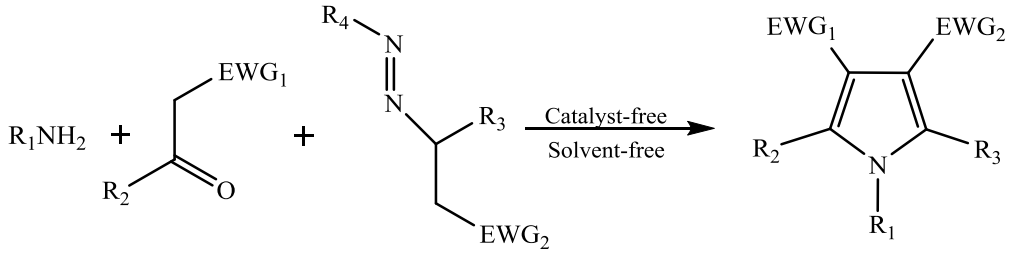
Scheme-1

Surya De[13] reported N-substituted pyrrole derivatives were synthesized by the reaction of hexane-2,5-dione, aniline and $Pr(OTf)_3$ as a catalyst under solvent-free conditions. The reaction was also tested in different solvents such as DCM, MeOH and THF and the best results were obtained in solvent-free neat conditions. (Scheme-2)



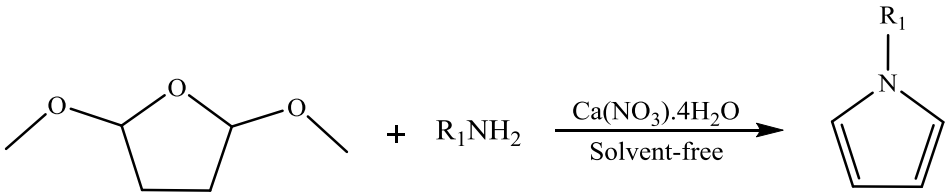
Scheme-2

Attanasi and coworkers[14] synthesized the polysubstituted pyrroles one-pot three component reaction between primary aliphatic amines,1,2-diaza-1,3-diene and active methylene compounds. The reaction was performed without using any catalyst and solvent. The mechanism involved the 3+2 cycloaddition of simple secondary enamines with 1,2-diaza-1,3-diene. (Scheme-3)



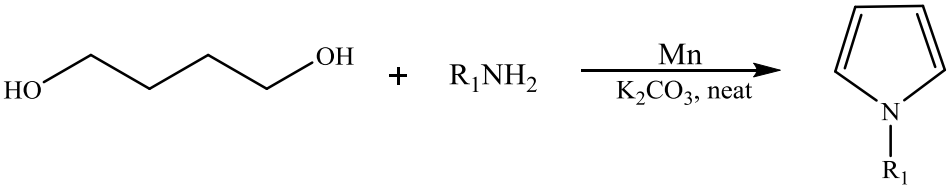
Scheme-3

Wani et al.[15] reported the synthesis of N-substituted pyrroles using alkaline earth metal-based catalyst. Reaction was carried out between 2,5-dimethoxytetrahydrofuran with aliphatic/aromatic amines and catalytic amount of $Ca(NO_3)_2.4H_2O$. It was observed that good to excellent yield of product was obtained solvent-free condition with the presence of electron donating groups on amines. (Scheme-4)



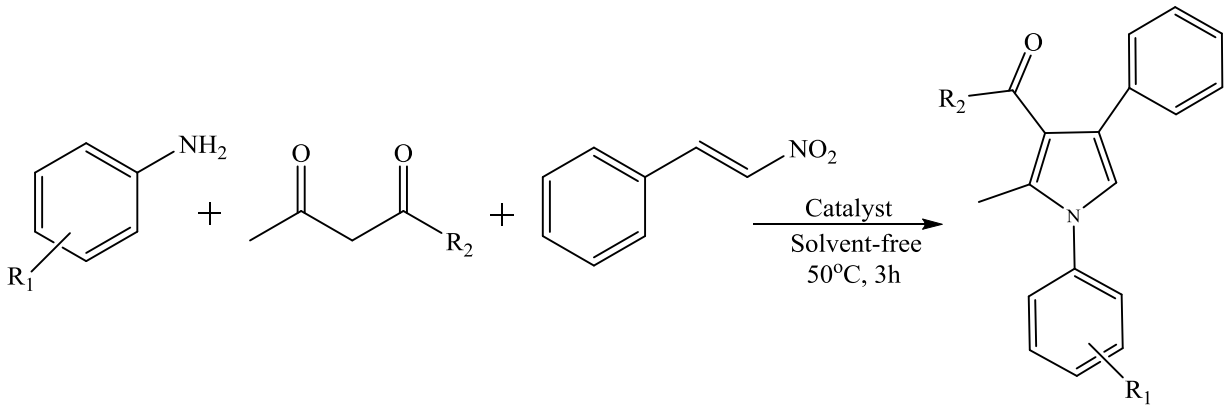
Scheme-4

Borghs and researchers[16] successfully prepared the 2,5-disubstituted pyrroles using biomass-derived primary diols and amines catalyzed by manganese/potassium carbonate under neat method which produces water and hydrogen gas as the byproducts. (Scheme-5)



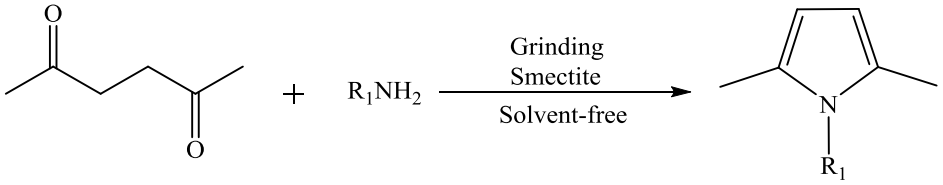
Scheme-5

Rostami and Shiri [17] developed an efficient one-pot multicomponent protocol for the synthesis of N-substituted pyrroles by using aniline derivatives, β-ketoesters/β-diketones and β-nitrostyrene catalyzed by Fe_3O_4@SiO_2–CPTMS–Guanidine–SO_3H under solvent-free conditions. The reactions were investigated at different temperatures and with the use of various polar solvents and the best results were obtained under solvent-free conditions at 50°C. (Scheme-6)



Scheme-6

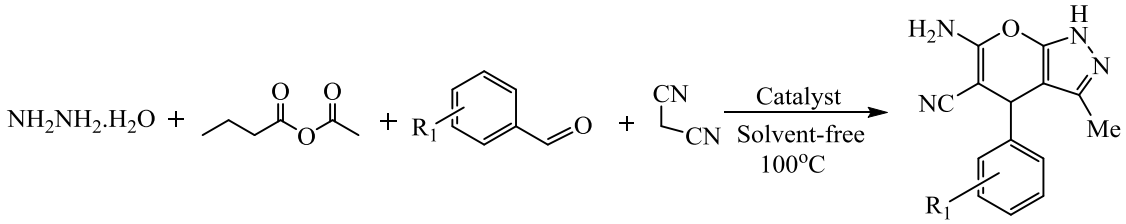
For the preparation of pyrrole derivatives, an environment-friendly neat procedure was reported by Marvi and Nahzomi[18] using Paal-Knorr condensation reaction between 2,5-hexandione and different aromatic amines catalyzed by smectite clays as heterogeneous Lewis acid catalysts. (Scheme-7)



Scheme-7

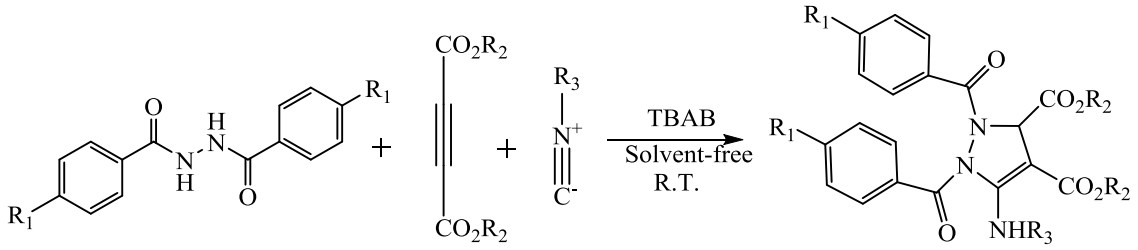
B. NEAT SYNTHESIS OF PYRAZOLES

Shaterian and Kangani [19] synthesized 1,4-dihydropyrano[2,3-c]pyrazoles using hydrazine monohydrate, ethyl acetoacetate, aryl aldehydes and malononitrile in the presence of P_2O_5/SiO_2, H_3PO_4/ Al_2O_3, cellulose sulfuric acid or starch sulfuric acid as a green catalyst under solvent free conditions. (Scheme-8)



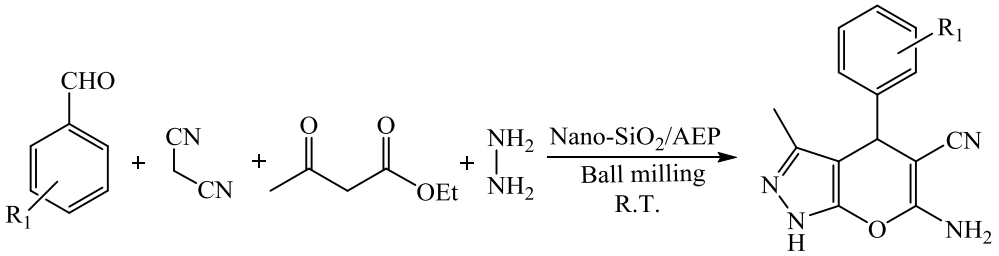
Scheme-8

Soltanzadeh et al.[20] described the synthesis of pyrazole derivatives under solvent free conditions. They used 4-phenylurazole or 1,2-dibenzoylhydrazines, dialkyl acetylene dicarboxylates, isocyanides and tetrabutylammonium bromide (TBAB) and obtained the product in good yield. (Scheme-9)



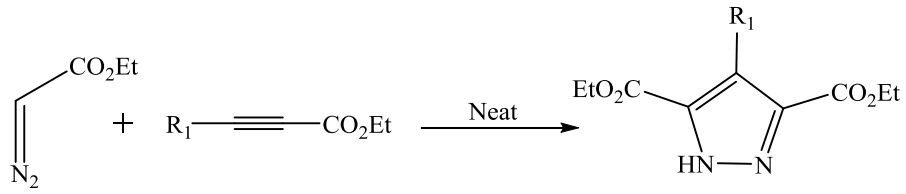
Scheme-9

Mallah and Mirjalili[21] reported the effective and green ball milling method for the synthesis of dihydropyrano[2,3-c]pyrazole using nano-silica/aminoethylpiperazine as a metal-free catalyst. They carried the reaction at room temperature and without using any solvent by obtaining the product in good to excellent yield. (Scheme-10)



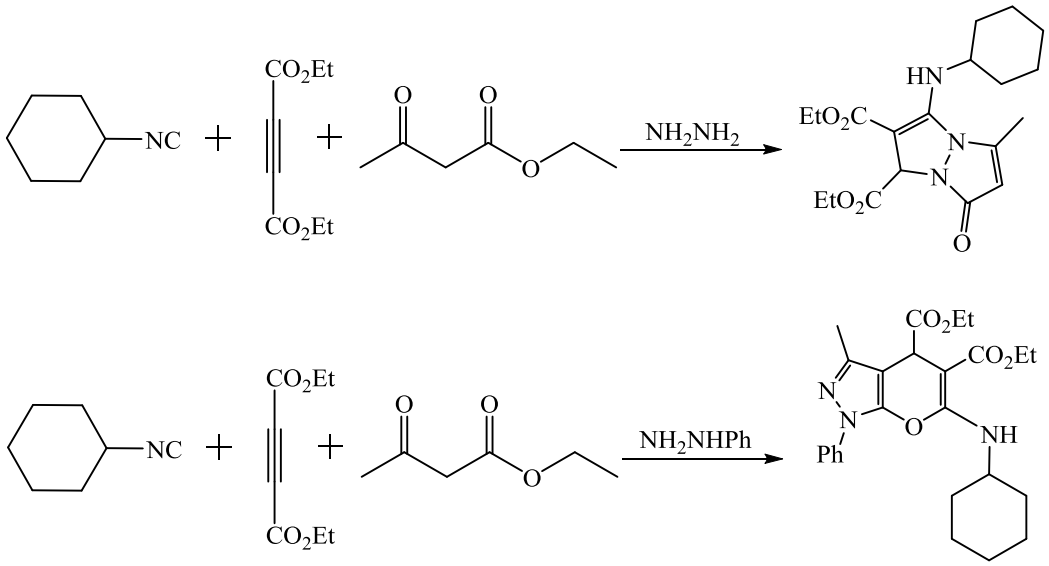
Scheme-10

Vuluga and coworkers[22] synthesized pyrazoles by 1,3-dipolar cycloaddition of diazo compounds and alkynes at 80°C under solvent-free conditions. They reported the method is simple and clean and the reaction affords pyrazoles in excellent yields. (Scheme-11)



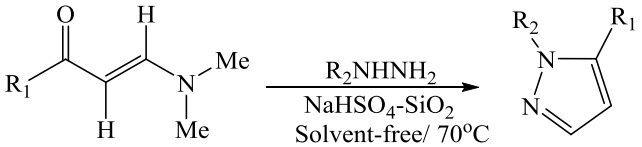
Scheme-11

Shaabani and co-researchers[23] reported the synthesis of Pyrazolo[1,2-a]Pyrazoles and Pyrano[2,3-c]Pyrazoles by using four component reaction between dialkyl acetylenedicarboxylates, isocyanides and ethyl acetoacetate with hydrazine hydrate or phenylhydrazine. They used different reaction conditions and concluded that they obtained the product in excellent yield under solvent-free conditions at 70°C. (Scheme-12)



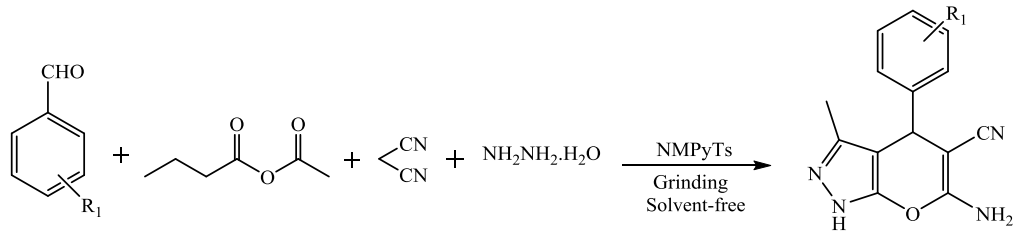
Scheme-12

Siddiqui and Farooq[24] reported the synthesis of substituted pyrazoles catalyzed by silica supported sodium hydrogen sulfate ($NaHSO_4.SiO_2$) under solvent-free conditions by the reaction of β-enaminones and hydrazine hydrate. (Scheme-13)



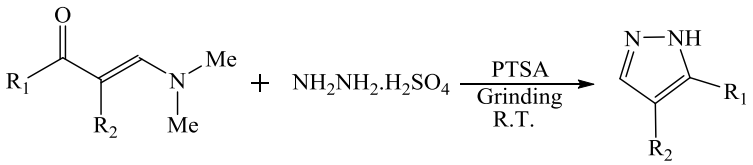
Scheme-13

Sapkal and researchers[25] reported an efficient and facile one pot multicomponent synthesis of Pyrano [2,3-c] pyrazoles in good to excellent yield using different substituted benzaldehyde, malononitrile, ethyl acetoacetate, hydrazine hydrate under solvent free condition using ionic liquid N-methyl pyridinium toluene sulfonate (NMPyTs) as a catalyst by grinding method. (Scheme-14)



Scheme-14

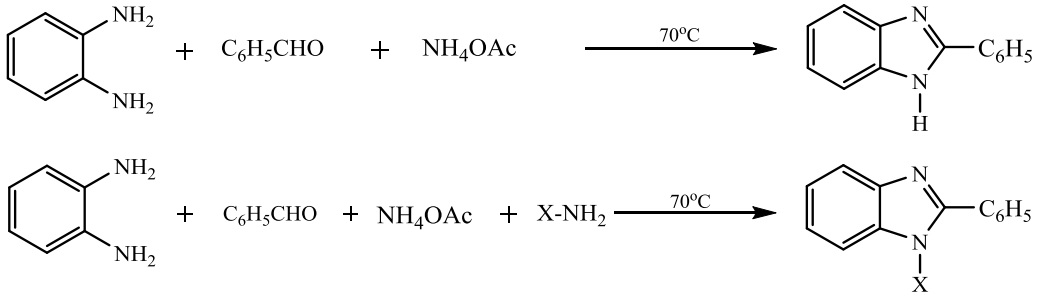
Longhi and team [26] synthesized the series of NH-pyrazoles using β-dimethylamino-vinyl ketones and hydrazine sulfate in solid state on grinding under solvent-free conditions. They used four different catalysts viz SiO_2, PTSA, $KHSO_4$, and $NaHSO_4$ and found PTSA (p-toluenesulfonic acid) as an efficient catalyst. (Scheme-15)



Scheme-15

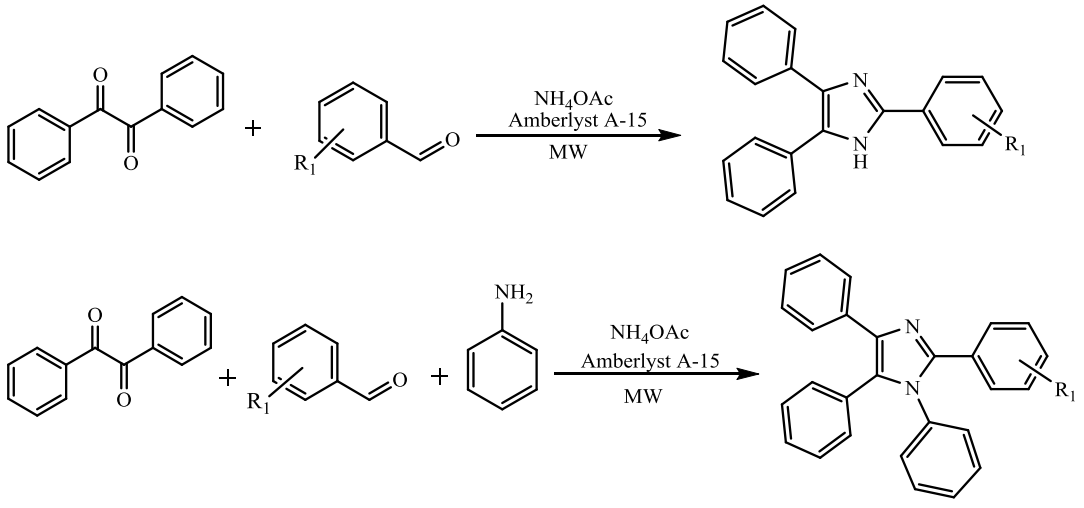
C. NEAT SYNTHESIS OF IMIDAZOLES

Zhang et al.[27] reported one-pot synthesis of imidazole derivatives using different substituted benzaldehyde, benzil or aromatic o-phenylenediamines and ammonium acetate under solvent-free conditions at 70°C and obtained the product in high yields. (Scheme-16)



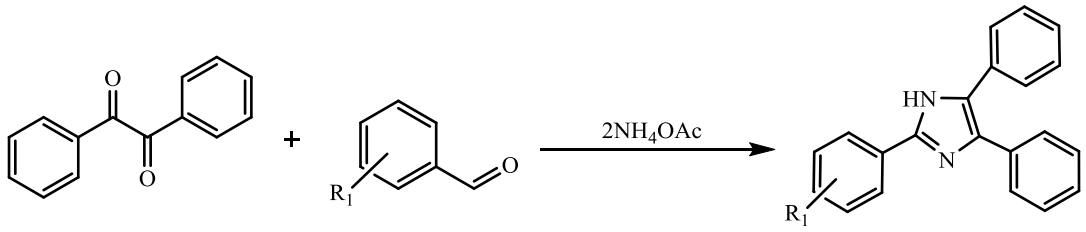
Scheme-16

The derivatives of 2,4,5-trisubstituted-1H-imidazole and 1,2,4,5-tetrasubstituted-imidazole were synthesized by Pandit and researchers[28] by benzyl, aryl aldehydes, ammonium acetate and aromatic amines in presence of Amberlyst A-15 as a reusable catalyst under microwave irradiations. (Scheme-17)



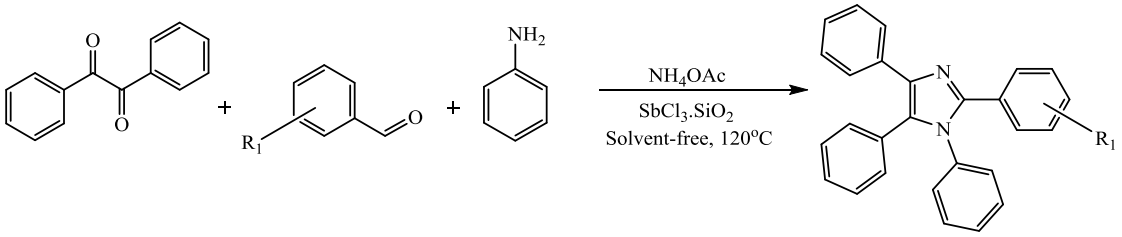
Scheme-17

Sangwan et al.[29] synthesized substituted imidazoles by reacting aromatic aldehydes, benzil, and ammonium acetate in presence of solid acid catalyst viz. Rice Husk Ash.SO_3H (RHA.SO_3H) at room temperature. They reported the synthesized products showed herbicidal activity against Raphanus sativus L. seeds and also screened for their antifungal activity against Rhizoctonia solani and Aspergillus niger. (Scheme-18)



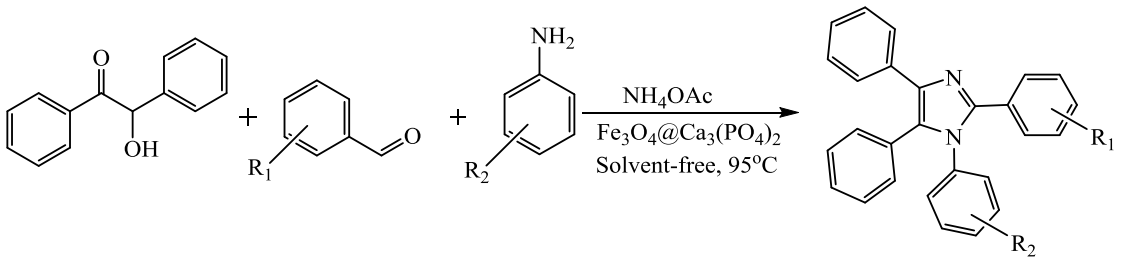
Scheme-18

Cyclocondensation of 1,2-diketone, aldehyde, ammonium acetate, primary amine and Antimony trichloride absorbed on silica gel ($SbCl_3/SiO_2$) as a catalyst under solvent-free conditions gives 1,2,3,5-tetrasubstituted imidazole derivatives as reported by Safari and team[30]. (Scheme-19)



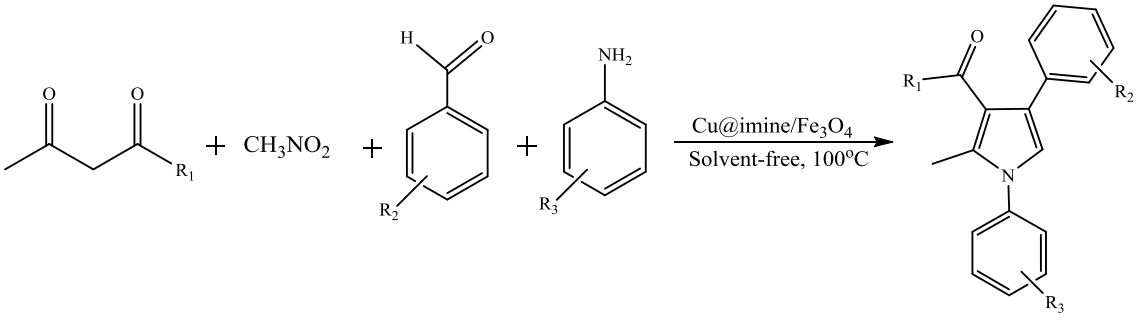
Scheme-19

Mahmoudiani et al.[31] prepared a new nano-Fe_3O_4@$Ca_3(PO_4)_2$ catalyst from an egg-shell as a solid waste with Fe_3O_4 nanoparticles. The synthesized nano-Fe_3O_4@$Ca_3(PO_4)_2$ catalyst was used as a promoter for the synthesis of 1,2,4,5-tetra-substituted imidazole derivatives by using various benzaldehydes, anilines, benzoin, and ammonium acetate at 95°C under solvent-free conditions. (Scheme-20)



Scheme-20

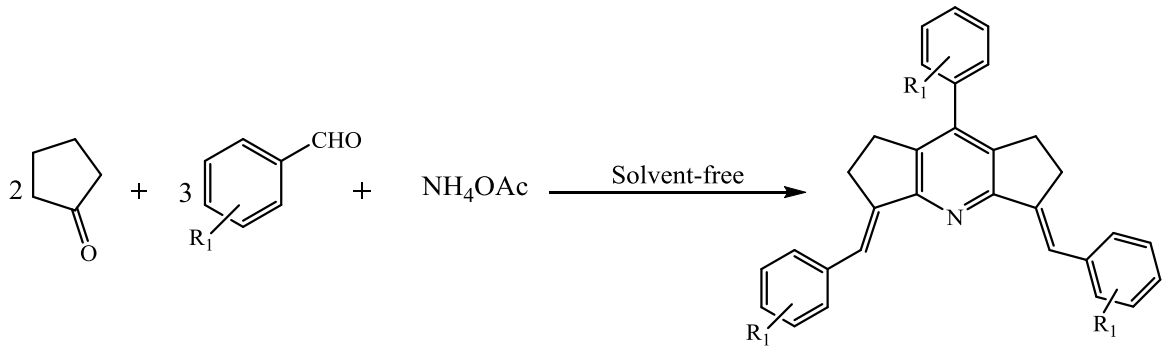
Thwin and researchers[32] reported the synthesis of 1,2,4,5-tetrasubstituted imidazole derivatives using benzil, benzaldehydes, benzalamines, and ammonium acetate in the presence of Cu@imine/Fe_3O_4 MNPs as a catalyst at 80°C under solvent-free conditions. (Scheme-21)



Scheme-21

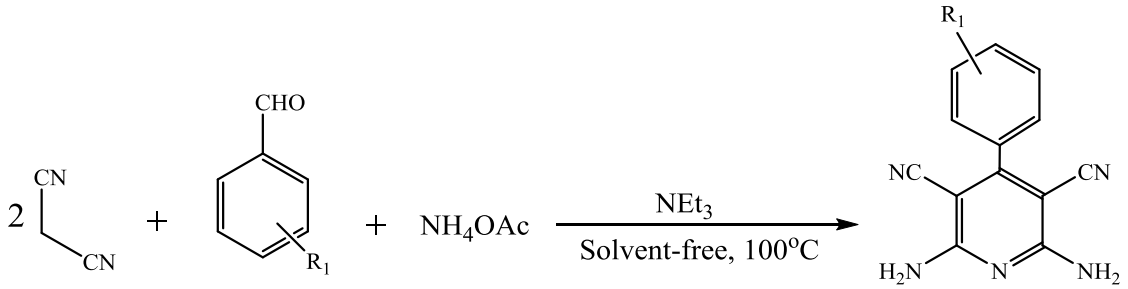
D. NEAT SYNTHESIS OF PYRIDINES

Rong and researchers[33] discussed a green protocol for the synthesis of polysubstituted pyridines without using any solvent and catalyst. The reaction was carried out between aromatic aldehydes, cyclopentanone and ammonium acetate and obtained the products in excellent yield. (Scheme-22)



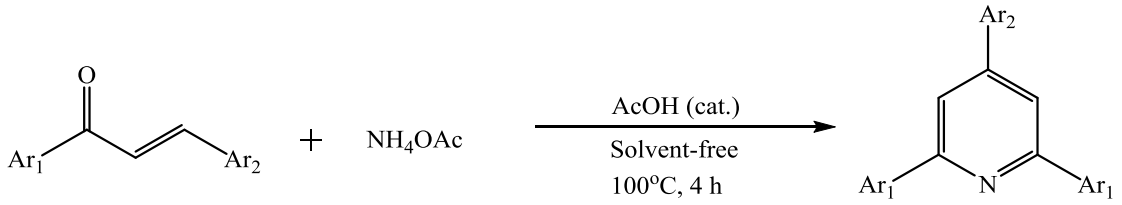
Scheme-22

Mobinikhaledi et al.[34] synthesized highly substituted pyridines by one-pot multicomponent reaction of aldehydes, malononitrile, and ammonium acetate in the presence of triethylamine(NEt_3) as a catalyst under solvent-free conditions. The reaction was investigated using various solvents and obtained the high yield of products under solvent free conditions at 100°C. (Scheme-23)



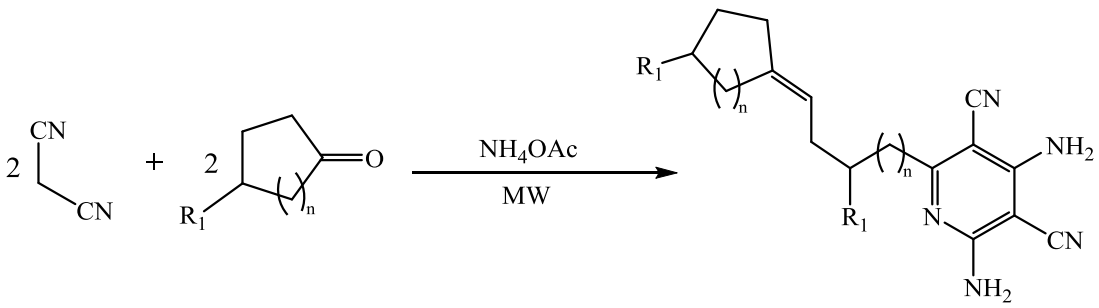
Scheme-23

Adib and coworkers[35] and researchers reported the novel method for synthesizing 2,4,6-triaryl pyridines through the reaction of 1,3-diaryl-2-propen-1-ones with ammonium acetate in the presence of a catalytic amount of acetic acid at 100°C for 4 hours, under solvent-free conditions. This method offers several advantages, including solvent-free conditions, high yields, and a simplified purification process. (Scheme-24)



Scheme-24

Jiang and researchers[36] reported a domino reaction of malononitrile, cycloketones and ammonium acetate under microwave-irradiation and solvent-free conditions to yield highly substituted 2,4-diaminopyridine-3,5-dicarbonitriles. The method was tested in different solvents but the product was obtained in excellent yield under solvent-free conditions. (Scheme-25)



Scheme-25

Conclusion

Neat reaction strategies, which eliminate the need for solvents and catalysts, have emerged as a vital tools in modern organic synthesis. This review has highlighted various neat reaction methods, including room-temperature reactions, microwave irradiation, grinding and ball milling. These approaches not only reduce waste and environmental impact but also offer simplicity, efficiency, and cost-effectiveness. This approach enables the straightforward synthesis of various compounds, including pyrroles, pyrazoles, imidazoles, and pyridines.

As the chemical industry focuses more on sustainability and protecting the environment, neat reaction strategies will become more important. Future research should work on improving these methods, finding new uses for them, and using them in large-scale industrial processes. In the end, using neat reaction strategies will help create chemical processes that are more efficient, sustainable, and better for the environment.

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