CHAPTER - IV

Synthesis and characterization of certain new Imidazoles and Benzimidazole compounds
4.1. Introduction

Imidazole ((CH)₂N(NH)CH) is a colourless diazole type aromatic heterocycle (alkaloid) solid that dissolves in water to give mildly alkaline solution. Derivatives of imidazole (imidazoles) form a common family of heterocycles that share the 1,3-C₃N₂ ring but feature varied substituents. This ring system is present in important biological building-blocks, such as histidine, and the related hormone histamine [1]. Many drugs contain an imidazole ring, such as antifungal drugs, nitro imidazole, and the sedative midazolam [2-6]. It forms purine when fused to a pyrimidine ring which is the most widely occurring nitrogen-containing heterocycle in nature [7]. Imidazole is a planar 5-membered ring and exists in two equivalent tautomeric forms, because the proton can be located on either of the two nitrogen atoms. Imidazole is a highly polar compound, as evidenced by a calculated dipole of 3.61D. It is highly soluble in water. The compound is classified as aromatic due to the presence of a sextet of π-electrons, consisting of a pair of electrons from the protonated nitrogen atom and one from each of the remaining four atoms of the ring. Some resonance structures of imidazole are shown below:

Imidazole is amphoteric which can function as both an acid and as a base. As an acid, the pKₐ of imidazole is 14.5, making it less acidic than carboxylic acids, phenols, and imides, but slightly more acidic than alcohols. The acidic proton is located on N-1. As a base, the pKₐ of the conjugate acid (cited above as pKᵦ⁺ to avoid confusion between the two) is approximately 7, making imidazole approximately sixty times more basic than pyridine. The basic site is N-3. Protonation gives the imidazolium cation, which is symmetrical. Imidazole was first reported in 1858, although various imidazole derivatives had been discovered as early as the 1840s. It is synthesized from used glyoxal and formaldehyde in ammonia to form imidazole (or glyoxaline, as it was originally named) [8].
Even though this synthesis produces relatively low yields, it is still used for creating C-substituted imidazoles. Imidazole can be synthesized by numerous methods besides the Debus method. Many of these syntheses can also be applied to different substituted imidazoles and imidazole derivatives by varying the functional groups on the reactants. In one microwave modification, the reactants are benzil, benzaldehyde and ammonia in glacial acetic acid, forming 2, 4,5-triphenylimidazole (Lophine) [9]. Van Leusan’s research work[10] revealed that base-induced cycloaddition of tosylmethylisocyanide (TosMIC) to aldimines in protic medium which occurs with subsequent elimination of p-toluene sulfonic acid to give the otherwise difficultly accessible 1,4-disubstituted imidazoles. Addition of TosMIC to imidoyl chlorides is accompanied by the loss of HCl, and leads to 1,4,5-trisubstituted imidazoles.

The reaction has later been expanded to a two-step synthesis in which the aldimines generated in situ.

Recent literature depicted a number of protocols comprising the synthesis of imidazoles and benzimidazoles [11-60]. Bratulescu enabled a simple and efficient microwave-assisted synthesis of 4, 5-disubstituted imidazoles[11] starting from 1, 2-diketones and urotropine in the presence of ammonium acetate under solvent free conditions.
Adib, Ansari and several others [12] achieved a one-pot, four-component synthesis of 1,2,4-trisubstituted 1H-imidazoles in very good yields by heating a mixture of a 2-bromoacetophenone, an aldehyde, a primary amine, and ammonium acetate under solvent-free conditions.

Zuliani and coworkers’ approach [13] allowed a simple and efficient preparation of biologically active 2, 4(5)-diarylimidazoles by parallel synthesis. The formation of 2-aryl-4(5)-arylimidazoles as side products strongly depends on the reaction conditions employed. Siddiqui et al [14] suggested an improved and rapid one-pot synthesis of 2, 4, 5-triaryl imidazoles in a room temperature ionic liquid does not need any added catalyst. This one-pot methodology offers excellent isolated yields, simple work up procedures and efficient recovery and recycling of the ionic liquid. Tang et al [15] developed a simple route via a copper-catalyzed [3 + 2] cycloaddition reaction provides multisubstituted imidazoles in good yields and high regioselectivity using oxygen as an oxidant without the addition of expensive catalysts.

The copper-catalyzed reaction between two different isocyanides produced imidazoles in good yields. In this protocol, mechanism of the reaction is also discussed by Kanazawa et al [16].

Horneff and co-workers [17] examined Rhodium (II)-catalyzed reaction of stable and readily available 1-sulfonyl triazoles with nitriles, which afforded corresponding imidazoles in good to excellent yields via rhodium iminocarbenoids intermediates.
Li and Neuville[18] developed copper (CuCl$_2$·2H$_2$O)-catalyzed regioselective diamination protocol with terminal alkynes and amidines in the presence of Na$_2$CO$_3$, pyridine, and oxygen (1 atm). The synthesis allowed diverse 1, 2, 4-trisubstituted imidazoles in good yields.

In Wang and coworkers’ research work [19], the use of a copper catalyst allows the construction of aryl imidazolium salts in good yields from $N$-substituted imidazoles and diaryliodonium salts. The reaction tolerates a broad range of functional groups and provides a straightforward, efficient, and versatile route to unsymmetric aryl imidazolium aswellastriazolium salts. Reactions of propargylamines with carbodiimides, in the presence of 5 mol% of the titanacarbonare monoamide $[$σ:η$^1$:η$^5$-OCH$_2$](Me$_2$NCH$_2$)C$_2$B$_9$H$_9$] Ti(NMe$_2$), performed by Wang, Shen and Xie [20] afforded a new class of substituted 2-aminoimidazoles via [3+2] annulation in good to excellent yields. A possible reaction mechanism is also proposed by the authors in this publication.

Hu, Wang and several others [21] developed a highly efficient and convenient method for the synthesis of 1, 2, 4, 5-tetrasubstituted imidazoles from readily accessible 2-azido acrylates and nitrones proceeded under mild conditions without the assistance of any metal, acid, or base.
Rajaguru et al. [22] examined a multi-component protocol which enabled the synthesis of highly substituted imidazole derivatives in excellent yield from various α-azidochalcones, aryl aldehydes, and anilines in the presence of erbium triflate as a catalyst.

In a gold-catalyzed synthesis of bicyclic imidazoles, Xiao, and Zhang [23] achieved a highly electrophilic α-imino gold carbene intermediate can react with a weakly nucleophilic nitrile, which is used as the reaction solvent, to deliver the desired product rapidly in an overall bimolecular [2 + 2 + 1] cycloaddition and in good yield. The competing intramolecular azide-alkyne click reaction, although likely also catalyzed by gold, is minimized by using AuCl₃ as the catalyst.

In a one-pot procedure for the conversion of aromatic and hetero aromatic 2-nitroamines into bicyclic 2H-benzimidazoles, Hanan et al. [24] employed formic acid, iron powder, and NH₄Cl as additive to reduce the nitro group and effect the imidazole cyclization with high-yielding conversions generally within one to two hours. The compatibility with a wide range of functional groups demonstrates the general utility of this procedure.
Nguyen et. al [25] used elemental sulfur as a traceless oxidizing agent under solvent-free and catalyst-free conditions for the synthesis of benzazoles from o-hydroxy/amino/mercaptanilines and alkylamines.

\[
\begin{align*}
\text{NH}_2 & + R''N^+R'^+ + 1.5\text{ eq.} \quad 3\text{ eq. sulfur} \quad 32\text{ g/mol} \\
\text{Ar} & \quad \text{neat} \quad 130^\circ C, 16\text{-}20\text{ h} \quad \text{Ar}
\end{align*}
\]

In another protocol, Nguyen’s research group [26] prepared a broad range of functionalized 2-aryl benzimidazoles a solvent-free cobalt- or iron-catalyzed redox condensation of 2-nitroanilines and benzylamines via benzylamine oxidation, nitro reduction, condensation, and aromatization without any reducing or oxidizing agent. The method can be extended to afford various other diazaheterocycles.

\[
\begin{align*}
\text{NH}_2 & + 3\text{ eq.} \quad 2\text{ to 5 mol}\% \quad 120\text{ or } 140^\circ C, 24\text{ h} \\
\text{Ar} & \quad \text{neat} \quad \text{Ar}
\end{align*}
\]

Mayo’s research reports [27] suggested that Brønsted acid catalyzed cyclization reactions of 2-amino thiophenols and anilines with β-diketones under oxidant- and metal-free conditions afforded 2-substituted benzothiazoles and benzimidazoles in good yields, respectively. Various groups such as methyl, chloro, nitro, and methoxy linked on benzene rings were tolerated under the optimized reaction conditions.

\[
\begin{align*}
\text{NH}_2 & + 1.5\text{ eq.} \quad 5\text{ mol}\% \quad \text{neat, r.t., 16 h or MeCN, } 80^\circ C, 16\text{ h} \\
\text{Ar} & \quad \text{Y}: \text{NH, S, } \text{R}: \text{Me, Et, iPr, Ph}
\end{align*}
\]

The reaction of ortho-substituted anilines with functionalized orthoesters yields benzoxazole, benzothiazole, and benzimidazole derivatives in an efficient and connective methodology. The versatility of this approach enabled Bastug and others [28] to develop new libraries of heterocycles containing multifunctional sites.
Caron, Jones and Wei [29] prepared benzimidazoles and imidazopyridines proceeds smoothly under mild conditions in isopropyl alcohol at 70°C using 2,2,2-trichloroethylimidates as the acylating agents. Addition of sodium acetate proved to be beneficial in cases where cyclization proceeded slowly. For substrates with poor nucleophilicity, using the more inert tert-amyl alcohol enabled superior reactions.

Bahrami, Khodaei, and Naali developed a convenient method [30] for the synthesis of 2-substituted benzimidazoles and benzothiazoles that offered short reaction times, large-scale synthesis, easy and quick isolation of the products, excellent chemo selectivity, and excellent yields as main advantages.

Kim, and coworkers [31] worked on copper-catalyzed, one-pot, three-component reaction of 2-haloanilines, aldehydes and NaN₃ enabled the synthesis of benzimidazoles in good yields using catalytic amounts of CuCl and TMEDA in DMSO at 120°C for 12 h. The reaction tolerated many functional groups such as ester, nitro, and chloro during this synthesis.
Chapter - IV

The research findings of Baars et. al [32] revealed intramolecular N-arylations of amidines mediated by potassium hydroxide in DMSO at 120°C enable the preparation of diversely substituted benzimidazoles in good yields.

![Diagram of chemical reaction]

Alla, Kumar and others used iodobenzene as a catalyst [33] for the synthesis of 1,2-disubstituted benzimidazoles by oxidative C-H amination of N''-aryl-N'-tosyl/N'-methylsulfonylamidines and N,N'-bis(aryl)amidines in the presence of MCPBA as terminal oxidant at room temperature. The reaction is general, and the target products can be obtained in good yields.

![Diagram of chemical reaction]

Diao, Wang, Jiang, and Ma [34] used CuI/proline as a catalyst for coupling of aqueous ammonia with 2-iodoacetanilides and 2-iodophenylcarbamates to afford aryl amination products at room temperature, which underwent in situ additive cyclization under acidic conditions or heating to give substituted 1H-benzimidazoles and 1,3-dihydrobenzimidazol-2-ones, respectively.

![Diagram of chemical reaction]

Punniyamurthy’s research group [35] developed an experimentally simple, general, efficient, and ligand-free synthesis of substituted benzimidazoles, 2-aminobenzimidazoles, 2-aminobenzothiazoles, and benzoxazoles via intramolecular cyclization of o-bromoaryl derivatives is catalyzed by copper (II) oxide nanoparticles in DMSO under aerobic conditions. The catalyst could be recovered and recycled without loss of activity.
In another successful research task, Punniyamurthy, Giru and Ali [36] developed an efficient method for the transformation of \( N \)-benzyl bisarylhydrazones and bisarylloxime ethers to functionalized 2-aryl-\( N \)-benzylbenzimidazoles and 2-arylbenzoxazoles involves a copper(II)-mediated cascade C-H functionalization/C-N/C-O bond formation under neutral conditions. Substrates having either electron-donating or withdrawing substituents undergo the cyclization at moderate temperature.

Ryabukhin et al. [37] readily prepared a set of benzimidazoles, 3\( H \)-imidazo[4,5-b]pyridines, purines, xanthines and benzothiazoles from (hetero)aromatic ortho-diamines or ortho-aminothiophenol and aldehydes using chlorotrimethylsilane in DMF as a promoter and water-acceptor agent, followed by oxidation with air oxygen.

Rosenberg, Zhao and Clark [38] successfully performed Pd-catalyzed amide coupling reaction for facile synthesis of imidazo[4,5-b]pyridines and -pyrazines. This reaction provides quick access to various substituted products. A model system relevant to the natural product pentosidine has been demonstrated, as well as the total synthesis of the mutagen 1-Me-5-PhIP.
Yang and others [39] were successful to develop a highly efficient and versatile method for the synthesis of a series of 2-substituted N-H, N-alkyl, and N-aryl benzimidazoles containing a wide range of functional groups was achieved in one step via the \( \text{Na}_2\text{S}_2\text{O}_4 \) reduction of \( o \)-nitroanilines in the presence of aldehydes. Bahrami, Khodaei and Kavianinia [40] established a simple and efficient procedure for the synthesis of substituted benzimidazoles through a one-pot condensation of \( o \)-phenylenediamines with aryl aldehydes in the presence of \( \text{H}_2\text{O}_2 \) and \( \text{HCl} \) in acetonitrile at room temperature. Other important features of this method included short reaction time, easy and quick isolation of the products, and excellent yields. Du and Wang [41] synthesized various 2arylbenzimidazoles from phenylenediamines and aldehydes via a one-step process using hypervalent iodine as oxidant. This method has the features such as mild conditions, short reaction times, high yields, and a simple work-up procedure. Beaulieu’s research group [42] revealed that addition of oxone to a mixture of a 1, 2-phenylenediamine and an aldehyde in wet DMF results in rapid formation of benzimidazoles under very mild conditions. Products are isolated in high purity in most cases by simple aqueous precipitation. The reaction is applicable to a wide range of substrates but does not allow the conversion of aldehydes that are sensitive to oxone under acidic reaction conditions. CuI and others’ mild and efficient one-pot synthesis [43] enabled the preparation of 2-substituted benzimidazoles from 1, 2-phenylenediamines and triacyloxyborane intermediates generated in situ from carboxylic acids and borane-THF. This protocol tolerates acid-labile functional groups. She, Jiang and Wang [44] examined efficient and general cascade reactions of \( o \)-aminoanilines or naphthalene-1,8-diamine with terminal alkynes and \( p \)-tolylsulfonylazide allow a one-pot synthesis of functionalized benzimidazoles and \( 1H \)-pyrimidines in good yields. Sluiter and Christoffers [45] established a \( \text{NaH} \)-mediated reaction of carbonitriles and \( N \)-methyl-1,2-phenylenediamine allows the formation of \( N \)-methyl benzimidazole and tolerates acid-labile acetal protective groups. These products were further converted in Suzuki, Sonogashira, Heck and Buchwald-Hartwig reactions. Peng et. al [46] suggested a fruitful, straightforward, efficient, and sustainable method for intramolecular \( N \)-arylation provides a library of benzimidazoles in high yields using \( \text{Cu}_2\text{O} \) as the catalyst, DMEDA as the ligand, and \( \text{K}_2\text{CO}_3 \) as the base. Remarkably, the reaction was exclusively carried out in water,
rendering the methodology highly valuable from both environmental and economical points of view. Wray and Stambuli [47] synthesized various N-aryl-1H-indazoles and benzimidazoles from common arylaminooximes in good to excellent yields depending upon the base used in the reaction. Triethylamine promoted the formation of benzimidazoles, whereas 2-aminopyridine promoted the formation of N-aryl imidazoles. Lv and Bao [48] were successful to promote. An efficient Cu(I)-catalyzed cascade intermolecular addition/intramolecular C-N coupling process enables the synthesis of a wide variety of 2-heterobenzimidazoles from o-haloarylcarbodiimides and N- or O-nucleophiles. Lukasik and Wróbel [49] performed condensation of N-aryl-2-nitrosoanilines with triphenylphosphine to give substituted aryliminophosphoranes, which furnished 2-alkylaminobenzimidazole derivatives in high yields in a subsequent reaction with alkyl isocyanates. Ishihara and Togo [50] prepared 2-Imidazolines in good yields from the reaction of aldehydes and ethylenediamine with iodine in the presence of potassium carbonate. The prepared 2-midazolines were smoothly oxidized to the corresponding imidazoles in good yields using (diacetoxyiodo)benzene at room temperature. Nicolaou et.al [51] discovered a number of new reactions of IBX with heteroatom-containing substrates were discovered and their utility was demonstrated. IBX was used for the generation of imines from secondary amines in notably high yields, for the oxidative aromatization of nitrogen heterocycles and for the cleavage of dithianes. Hirano’s research group developed a versatile and modular one-pot synthetic method [52] for the preparation of differently substituted symmetrical and unsymmetrical imidazolium salts from readily available form amidines and α-halo ketones. For many substitution patterns of the imidazolium salt products, this efficient strategy compares favorably with well-known processes in terms of yield, ease of synthesis, and robustness. Zhang and others [53] achieved CuI-catalyzed aerobic oxidative synthesis of imidazo[1,2-α]pyridines from 2-aminopyridines and acetophenones is compatible with a broad range of functional groups. The reaction also enabled the formation of alkenyl-substituted imidazo heterocycles using unsaturated ketones as substrates. Preliminary mechanistic studies indicate that this reaction proceeds through a catalytic Ortoleva-King reaction. Huang et. al [54] accomplished a rapid, copper-catalyzed aerobic dehydrogenative cyclization of pyridines with ketone oxime esters enables an environmentally friendly
synthesis of imidazo[1,2-a]pyridines. Yan et al. accomplished iron-catalyzed denitration reaction that enabled the synthesis of 3-methyl-2-arylimidazo[1,2-a]pyridine derivatives in good yields from aminopyridines and 2-methyl-nitroolefins. The procedure is simple and inexpensive and tolerates various functional groups. Mohan, Rao, and Adimurthy examined aqueous syntheses gives methylimidazo[1,2-a]pyridines, imidazo[1,2-a] pyrazines, and imidazo[2,1-a]isoquinolines without any deliberate addition of catalyst. On the other hand, using acetonitrile as solvent, Ag-catalyzed intramolecular aminooxigenation produced imidazo[1,2-a]pyridine-3-carbaldehydes in good yields. With a mixed Cu(I)-Cu(II) system in situ generated by partial reduction of CuSO₄ with glucose, an efficient and eco-friendly multicomponent cascade reaction of A³-coupling of heterocyclic amidine with aldehyde and alkyne, 5-exo-digcycloisomerization, and prototropic shift has afforded therapeutically important versatile N-fused imidazoles. Mishra and Ghosh achieved a one-pot reaction of aldehydes, 2-aminopyridines, and terminal alkynes, in the presence of the Copper(I) iodide-CuI-NaHSO₄-SiO₂ combination catalyst in refluxing toluene, generates the corresponding imidazo[1,2-a]pyridines in high to excellent yields. Adib et al. prepared N-Phenacylpyridinium bromides in situ from the addition of pyridines to α-bromoketones, which undergo nucleophilic addition of ammonium acetate under microwave irradiation and solvent-free conditions to afford the corresponding imidazo[1,2-a]pyridines in excellent yields. A simple and efficient protocol, developed by Wu, Pan and Zhou enabled the synthesis of 3-arylimidazo[1,2-a]pyridines by a catalyst-free cascade process from 2-aminopyridine and 1-bromo-2-phenylacetylene or 1,1-dibromo-2-phenylethene in yields up to 86%. Experiments of Yan et al. revealed that a metal-free sequential dual oxidative amination of C(sp³)-H bonds under ambient conditions affords imidazo[1,5-a]pyridines in very good yields. The reaction involves two oxidative C-N couplings and one oxidative dehydrogenation process with six hydrogen atoms removed. A facile formation of C-N, C-O, and C-S bonds from yynals, pyridin-2-amines, and alcohols or thiols enables a transition-metal-free three-component reaction for the construction of imidazo[1,2-a] pyridines. Yan et al. established a copper-catalyzed one-pot procedure which enabled the synthesis of imidazo[1,2-a]pyridines with aminopyridines and nitroolefins using air as oxidant. This general reaction appears to be
very suitable for the construction of various imidazo[1,2-\(a\)]pyridines. In a regioselective and high-yielding Groebke-Blackburn-Bienaymé reaction, glyoxallic acid is used as formaldehyde equivalent leading to a regioselective, mild, convenient, and effective synthesis of 3-aminoalkyl imidazoazines [64]. An efficient microwave-assisted metal-free amino benzannulation of aryl(4-aryl-1-(prop-2-ynyl)-1H-imidazol-2-yl) methanone with dialkylamines affords various 2,8-diaryl-6-aminoimidazo[1,2-\(a\)]pyridines in good yield [65].

Hutt and Aron[66] studied an efficient three-component coupling reaction of substituted picolin aldehydes, amines, and formaldehyde, which produced imidazo[1,5-\(a\)]pyridinium ions in high yields under mild conditions, allowing the incorporation of diverse functionality and chiral substituents. Higher order condensations are also described that provide access to multidentate NHC ligands useful for a variety of applications.

Chen and coworkers [67] established copper(I) bromide as an efficient catalyst for \(N\)-arylation of azoles with a variety of aromatic bromides and iodides under mild conditions displayed great functional group compatibility and excellent reactive selectivity.

Chen et al. [68] designed copper-catalyzed \(N\)-arylation reaction of imidazole proceeds under very mild conditions in the absence of additional ligand. This protocol tolerates an array of thermally sensitive functional groups, but also achieves high chemoselectivity.

Sreedhar et al. [69] efficiently carried out \(N\)-Arylation of azoles and amines with arylboronic acids using heterogeneous copper(I) oxide as catalyst in methanol at room temperature under base-free conditions. Various arylboronic acids and amines were
converted to the corresponding \(N\)-arylazoles and \(N\)-arylamines in very good yields, demonstrating the versatility of the reaction.

\[
\begin{align*}
\text{Ar}-\text{OH}_2 + \text{HN} & \xrightarrow{1,2 \text{ eq.}} \text{Ar}-\text{N} \quad \text{(5-5 eq. CuO)} \\
\text{MeOH, r.t., 5-15 h} & \quad \text{MeOH, r.t., 5-15 h}
\end{align*}
\]

Wang, Wu, and Shang [70] proposed a simple and efficient method enables the synthesis of \(N\)-alkynylheteroarenes from 1, 1-dibromo-1-alkenes via a copper-catalyzed cross-coupling reaction. Good yields and functional-group tolerance were obtained with TMEDA as ligand using imidazole and benzimidazole substrates in dioxane.

\[
\begin{align*}
\text{N} & \xrightarrow{5 \text{ eq. CuI}} \text{N} \\
\text{R} & \xrightarrow{0.1 \text{ eq. TMEDA}} \text{R} \\
\text{Br} & \xrightarrow{4 \text{ eq. Cs}_2\text{CO}_3} \text{Br} \\
\text{dioxane, 80° C, 24 h} & \quad \text{dioxane, 80° C, 24 h}
\end{align*}
\]

A series of \(N, N'\)-asymmetrically substituted imidazolium iodides have been synthesized by Oertel and co-workers [71] starting from \(N\)-arylimidazoles and the less expensive, but less reactive, 1-chlorobutane or (3-chloropropyl) trimethoxysilane. The addition of potassium iodide and the use of 1, 2-dimethoxyethane as a solvent allowed the synthesis of multigram quantities of these salts.

\[
\begin{align*}
\text{Ar} & \xrightarrow{1-1.7 \text{ eq. KI}} \text{Ar} \\
\text{N} & \xrightarrow{1 \text{ eq. LME, 75 or 85° C}} \text{N} \\
\text{Cl} & \xrightarrow{18-60 h} \text{Cl} \\
\text{Si(CMe)_3} & \xrightarrow{1 \text{ eq. LME, 75 or 85° C}} \text{Si(CMe)_3}
\end{align*}
\]

Liu et. al used \(N\)-Methylimidazole is a promising catalyst for aza-Michael reactions [72]. Various \(N\)-heterocycles were introduced to \(\alpha, \beta\)-unsaturated carbonyl compounds employing \(N\)-methylimidazole in a highly efficient, rapid and high yielding synthesis of \(N\)-heterocyclic derivatives.
2-Lithioimidazole was prepared by Torregrosa et. al [73] using lithium metal in the presence of a catalytic amount of isoprene in THF at room temperature. By reacting this organolithium compound with carbonyl electrophiles 2-(hydroxyalkyl) imidazoles and 2-(aminoalkyl)imidazoles were obtained in good yields.

Encouraged by the forgoing versatile literature reports on imidazoles and benzimidazoles, the author has taken up the present study to synthesize certain imidazole and benzimidazoles derivatives and there by test them for biological activities.

4.2. Synthesis and characterization of 2-(4-(2-phenylimidazo[1,2-a]pyridin-6-yl)phenyl)-1H-benzo[d]imidazole derivatives.

The methodology involved three steps Viz., (i) Synthesis of 6-bromo-2-phenylimidazo[1,2-a]pyridine , (ii) 4-(2-phenylimidazo[1,2-a]pyridin-6-yl) benzaldehyde and finally Synthesis of 2,6-diphenylimidazo[1,2-a] pyridine derivatives (III a to III e). Synthesis of 2,6-diphenylimidazo[1,2-a] pyridine derivatives (III -a to III-e) have been achieved by Suzuki reaction from4-(2-phenylimidazo[1,2-a]pyridin-6-yl)benzaldehyde (II a to II e) and substituted orthophenylenediamine at about 90°C under stirred conditions.

Step-1: Synthesis of 6-bromo-2-phenyl imidazo [1, 2-a]pyridine:
2-amino-5-bromo-pyridine and 2-bromo acetophenone was taken in a round bottom flask. To this add ethanol and reflux for about 4 hours. The reaction mixture was monitor by
Chapter - IV

TLC. After completion reaction solid precipitated and filtered. Wash with ethanol and dried.

Step-2: Synthesis of 2,6-diphenylimidazo[1,2-a]pyridine:

The 6-bromo-2-phenylimidazo[1,2-a]pyridine (0.25 g, 0.9 mmol, 1eq) and Boronic acid (0.12 g, 1.0 mmol, 1.1 eq.) was stirred in the presence of 4 mol% of tetrakis(triphenylphosphine) palladium (0) and Na₂CO₃ as a base at room temperature under nitrogen atmosphere for 30 min in DMF (15 mL). After 30 min, the reaction mixture was heated at 80°C for 12 hours, the progress of the reaction monitor by TLC. After completion of the reaction it was cooled to room temperature and diluted with water (50 mL). The aqueous layers were extracted with Ethyl acetate. The organic layer extracted, dried over Na₂SO₄ and the solvent evaporated. The residue was purified by column chromatography using EtOAc : hexane to give 2,6-diphenylimidazo[1,2-a]pyridine (II a to II f).
Entry | Product                                                                 | Yield (%) |
--- | ------------------------------------------------------------------------ |-----------|
II a | H=[2,6-diphenylimidazo[1,2-a]pyridine]                                   | 85        |
II b | 4-F=[6-(4-fluorophenyl)-2-phenylimidazo[1,2-a]pyridine]                  | 78        |
II c | 2-OCH₃=6-(2-methoxyphenyl)-2-phenylimidazo[1,2-a]pyridine                | 83        |
II d | 2-CH₃=[2-phenyl-6-(o-tolyl)imidazo[1,2-a]pyridine]                       | 88        |
II e | 2-OCH₃, 5-F=[6-(2-fluoro-6-methoxyphenyl)-2-phenylimidazo[1,2-a]pyridine] | 81        |
II f | 4-CHO=[4-(2-phenylimidazo[1,2-a]pyridin-6-yl)benzaldehyde]               | 90        |

Reaction Conditions: Pd(PPh₃)₄ (catyst)/Na₂CO₃/DMF (solvent) Reflux et 80-90 °C.
Table 4.1 Spectroscopic data for imidazole compounds (IIa – IIf)

<table>
<thead>
<tr>
<th>Entry</th>
<th>Physical and Spectroscopic data</th>
</tr>
</thead>
<tbody>
<tr>
<td>II a</td>
<td>(^1H) NMR (400 MHz, CDCl(_3)) (\delta ): 5.23 (s, 2H), 5.89 (s, 2H), 7.23 (d, J=9.0 Hz, 2H), 7.46 (m, 3H), 7.48 (m, 3H), 7.93 (s, 1H), 8.02 (ddd, J=1.2 Hz, J=8.0 Hz, J=9.5 Hz, 2H). M.W.: (M+1); 271.3</td>
</tr>
<tr>
<td>II b</td>
<td>(^1H) NMR (400 MHz, CDCl(_3)) (\delta ): 7.46 (t, J=8.5 Hz, 2H), 7.33 (m, 2H), 7.49 (m, 4H), 7.65 (d, J=2.0 Hz, 1H), 7.82 (s, 1H), 7.96 (d, J=7.0 Hz, 2H), 8.16 (s, 1H). M.W.: (M+1); 289.2</td>
</tr>
<tr>
<td>II c</td>
<td>(^1H) NMR (400 MHz, CDCl(_3)) (\delta ): 3.87 (s, 3H), 6.95 (dd, J=8.0 Hz, 1H), 7.09 (t, J=2.0 Hz, 3.87 Hz, 1H), 7.15 (d, J=7.5 Hz, 1H), 7.46 (m, 5H), 7.69 (d, J=7.5 Hz, 1H), 7.89 (s, 1H), 7.97 (d, J=7.2 Hz, 2H), 8.27 (s, 1H). M.W.: (M+1); 301.4</td>
</tr>
<tr>
<td>II d</td>
<td>(^1H) NMR (400 MHz, CDCl(_3)) (\delta ): 2.32 (s, 3H), 7.18 (dd, J=1.7 Hz, J=9.2 Hz, 1H), 7.36 (m, 5H), 7.47 (t, J=7.2 Hz, 15.0 Hz, 1H), 7.66 (d, J=9.2 Hz, 2H), 7.88 (s, 1H), 7.98 (d, J=7.0 Hz, 2H), 8.01 (bs, 1H). M.W.: (M+1); 285.2</td>
</tr>
<tr>
<td>II e</td>
<td>(^1H) NMR (400 MHz, CDCl(_3)) (\delta ): 3.83 (s, 3H), 6.88 (m, 1H), 7.14 (t, J=9.5 Hz, J=9.0 Hz, 1H), 7.37 (t, J=9.0 Hz, J=15.5 Hz, 2H), 7.46 (t, J=7.5 Hz, J=15.3 Hz, 2H), 7.69 (d, J=9.2 Hz, 1H), 7.89 (s, 1H), 7.98 (d, J=7.5 Hz, 2H), 8.33 (s, 1H). M.W.: (M+1); 319.3</td>
</tr>
<tr>
<td>II f</td>
<td>(^1H) NMR (400 MHz, CDCl(_3)) (\delta ): 7.38 (m, 1H), 7.49 (dd, J=10.3 Hz, J=11.2 Hz, 2H), 7.77 (m, 3H), 8.01 (m, 5H), 8.40 (s, 1H), 10.08 (s, 1H). M.W.: (M+1); 299.2</td>
</tr>
</tbody>
</table>

Step-3: Synthesis of 2-(4-(2-phenyl imidazo[1,2-a]pyridin-6-yl)phenyl)-1H-benzo[d]imidazole:

To a mixture of 4-(2-phenylimidazo [1, 2-a] pyridin-6-yl) benzaldehyde (0.250 g, 0.83 mol) and substituted ortho phenylene diamine (0.135, 1.25 mmol) were taken into DMF. To this add catalytic amount of sodium bisulphate (0.1eq) in to the mixture. The reaction mixture is refluxed for about 5-6 hrs at 90°C with constant stirring. After completion reaction was monitored by TLC, the reaction mixture is poured into crushed ice, filtered, and the resultant compound is dried. After recrystallization with methanol, pure compound is obtained. The prepared compounds are characterized by spectroscopic measurements. (III a to III e).
Chapter - IV

4-(2-phenylimidazo[1,2-a]pyridin-6-yl)benzaldehyde

\[ \text{R}_1 = \text{H}_2\text{NO}_2 \text{H-Br-Cl 3,4-di Cl} \]

\[ \text{(II)} \]

\[ \text{CHO} \]

\[ \text{H}_2\text{N-Br} \]

\[ \text{H}_2\text{N-H} \]

\[ \text{H}_2\text{N-N} \]

\[ \text{CHO} \]

\[ \text{H}_2\text{N-Br} \]

\[ \text{H}_2\text{N-H} \]

\[ \text{H}_2\text{N-N} \]

\[ \text{CHO} \]

\[ \text{H}_2\text{N-Br} \]

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\[ \text{H}_2\text{N-N} \]

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\[ \text{H}_2\text{N-Br} \]

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\[ \text{H}_2\text{N-Br} \]

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\[ \text{H}_2\text{N-Br} \]

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\[ \text{H}_2\text{N-Br} \]

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\[ \text{H}_2\text{N-Br} \]

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\[ \text{H}_2\text{N-N} \]

\[ \text{CHO} \]

\[ \text{H}_2\text{N-Br} \]

\[ \text{H}_2\text{N-H} \]

\[ \text{H}_2\text{N-N} \]

\[ \text{CHO} \]

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\[ \text{H}_2\text{N-N} \]

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\[ \text{H}_2\text{N-Br} \]

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\[ \text{H}_2\text{N-Br} \]

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\[ \text{CHO} \]

\[ \text{H}_2\text{N-Br} \]

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\[ \text{H}_2\text{N-Br} \]

\[ \text{H}_2\text{N-H} \]

\[ \text{H}_2\text{N-N} \]

\[ \text{CHO} \]

\[ \text{H}_2\text{N-Br} \]

\[ \text{H}_2\text{N-H} \]

\[ \text{H}_2\text{N-N} \]

\[ \text{CHO} \]

\[ \text{H}_2\text{N-Br} \]

\[ \text{H}_2\text{N-H} \]

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\[ \text{CHO} \]

\[ \text{H}_2\text{N-Br} \]

\[ \text{H}_2\text{N-H} \]

\[ \text{H}_2\text{N-N} \]

\[ \text{CHO} \]

\[ \text{H}_2\text{N-Br} \]

\[ \text{H}_2\text{N-H} \]

\[ \text{H}_2\text{N-N} \]
Table 4.2: Tabular form of III a–III e

<table>
<thead>
<tr>
<th>Entry</th>
<th>Substituent</th>
<th>Conventional</th>
<th>Sonication</th>
<th>Melting point(°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>R.T. (hr)</td>
<td>Yield (%)</td>
<td>R.T. (min)</td>
</tr>
<tr>
<td>III a</td>
<td>H=[ 2-(4-(2-phenylimidazol[1,2-a]pyridin-6-yl)phenyl]-1H-benzo[d]imidazole]</td>
<td>5</td>
<td>92</td>
<td>10</td>
</tr>
<tr>
<td>III b</td>
<td>4-Cl=[ 6-chloro-2-(4-(2-phenylimidazol[1,2-a]pyridin-6-yl)phenyl]-1H-benzo[d]imidazole]</td>
<td>5.5</td>
<td>85</td>
<td>15</td>
</tr>
<tr>
<td>III c</td>
<td>4-NO₂= 6-nitro-2-(4-(2-phenylimidazol[1,2-a]pyridin-6-yl)phenyl]-1H-benzo[d]imidazole]</td>
<td>6</td>
<td>82</td>
<td>15</td>
</tr>
<tr>
<td>III d</td>
<td>4,5-di Cl = 5,6-dichloro-2-(4-(2-phenylimidazol[1,2-a]pyridin-6-yl)phenyl]-1H-benzo[d]imidazole]</td>
<td>5</td>
<td>86</td>
<td>15</td>
</tr>
<tr>
<td>III e</td>
<td>4-Br=[ 6-bromo-2-(4-(2-phenylimidazol[1,2-a]pyridin-6-yl)phenyl]-1H-benzo[d]imidazole]</td>
<td>4</td>
<td>94</td>
<td>20</td>
</tr>
</tbody>
</table>

Table 4.3 Spectroscopic data for imidazole compounds (III a – III e)

<table>
<thead>
<tr>
<th>Entry</th>
<th>Physical and Spectroscopic data</th>
</tr>
</thead>
<tbody>
<tr>
<td>III a</td>
<td>'H NMR (400 MHz, DMSO): δ 9.04 (s, 1H), 8.43 (s, 1H), 8.33 (d, J=8.2 Hz, 2H), 8.02 (d, J=7.2 Hz, 2H), 7.96 (d, J=8.2 Hz, 2H), 7.72 (s, 2H), 7.63(m, 2H), 7.49 (t, J=7.5 Hz, J=15.0 Hz, 2H), 7.35(m, 1H), 7.24(m, 2H), M.W.(M+1): 387.1</td>
</tr>
<tr>
<td>III b</td>
<td>'H NMR (400 MHz, DMSO): δ 9.21 (s, 1H), 8.60 (s, 1H), 8.32 (m, 3H), 8.04 (m, 3H), 7.90 (t, J=8.2 Hz, 2H), 7.72 (s, 1H), 7.63(d, 1H), 7.55 (t, J=7.5 Hz, J=15.0 Hz, 2H), 7.44(m, 1H), 7.28(d, J=8.6Hz, 1H), 5.02(bs, 1H), M.W.(M+1): 421.1</td>
</tr>
<tr>
<td>III c</td>
<td>'H NMR (400 MHz, DMSO): δ 9.01 (s, 1H), 8.36 (s, 1H), 8.28 (d, J=8.1 Hz, 2H), 7.86 (s, 1H), 7.92(d, J=8.1 Hz, 2H), 7.68 (m, 2H), 7.56(s, 1H), 7.45 (t, J=15.0 Hz, J=7.5 Hz, 2H), 7.28(m, 1H), 7.21(m, 2H), M.W.(M+1): 432.5.</td>
</tr>
<tr>
<td>III d</td>
<td>'H NMR (400 MHz, DMSO): δ 9.19 (s, 1H), 8.61 (s, 1H), 8.31 (d, J=7.6 Hz, 2H), 8.05 (d, J=8.0 Hz, 2H), 7.97(m, 3H), 7.68 (m, 2H), 7.56(s, 1H), 7.45 (t, J=15.0 Hz, J=7.5 Hz, 2H), 7.28(m, 1H), 7.21(m, 2H), M.W.(M+1): 455.0.</td>
</tr>
<tr>
<td>III e</td>
<td>'H NMR (400 MHz, DMSO): δ 9.20 (s, 1H), 8.61 (s, 1H), 8.30 (d, J=8.3 Hz, 2H), 8.06 (d, J=9.6 Hz, 1H), 7.97 (m, 4H), 7.89 (d, J=9.3 Hz, 1H), 7.82 (m, 1H), 7.61 (m, J=8.6 Hz, 1H), 7.54 (t, J=15.1 Hz, J=7.5 Hz ,2H), 7.49(t, J=15.0 Hz, J=7.5 Hz 2H), 7.38(dd, J=1.5 Hz, J=1.8 .1H) M.W.;(M+1): 466.9</td>
</tr>
</tbody>
</table>
4.3. Ultrasonically assisted organic synthesis: It is interesting to note that the reactions are dramatically from 5-7 hrs to 10-20 min when conducted in ultrasonic sonicator. This observation could be well explained due to ultrasonic cavitation effect [74-76]. High-power ultrasound can generate cavitation within a liquid and through cavitation provide a source of energy which can be used to enhance a wide range of chemical processes. Cavitation is a process in which mechanical activation destroys the attractive forces of molecules in the liquid phase. Applying ultrasound, compression of the liquid is followed by rarefaction (expansion), in which a sudden pressure drop forms small, oscillating bubbles of gaseous substances. These bubbles expand with each cycle of the applied ultrasonic energy until they reach an unstable size; they can then collide and/or violently collapse. Energy thus released due to cavitation could be used for activation of molecules and thereby causing rate accelerations. Another such specific effect due to cavitation is the asymmetric collapse near a solid surface, which forms micro jets. This effect is the reason why ultrasound is very effective in cleaning, and is also responsible for rate acceleration in multiphasic reactions, since surface cleaning and erosion lead to improved mass transport.

4.4. Biological Activity Studies of Imidazole derivatives

Antibacterial activity and minimum inhibitory concentration (MIC) of the prepared 2-(4-(2-phenylimidazo[1,2-a]pyridin-6-yl)phenyl)-1H-benzo[d]imidazole (III a to III e) were determined against one gram-positive bacterium (Staphylococcus aureus) and one gram-negative bacterium (Pseudomonas aeruginosa) according to standard methods, as detailed in Chapter-II. Among the five different triazole derivatives, III d and III e have been found to exhibit maximum activity, while other derivatives exhibited marginal
activity with reference to CIPROFLOXACIN and GENTAMICIN, as shown in the following table.

<table>
<thead>
<tr>
<th>Imidazole</th>
<th>Pseudomonas aeruginosa MIC Value (µg/ml)</th>
<th>Streptococcus pyogenes MIC Value (µg/ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>III a</td>
<td>8.5</td>
<td>7.5</td>
</tr>
<tr>
<td>III b</td>
<td>10.0</td>
<td>7.5</td>
</tr>
<tr>
<td>III c</td>
<td>10.0</td>
<td>9.5</td>
</tr>
<tr>
<td>III d</td>
<td>8.5</td>
<td>9.5</td>
</tr>
<tr>
<td>III e</td>
<td>9.5</td>
<td>9.8</td>
</tr>
<tr>
<td>CIPROFLOXACIN</td>
<td>8.08</td>
<td>8.12</td>
</tr>
<tr>
<td>GENTAMICIN</td>
<td>2.5</td>
<td>6.5</td>
</tr>
</tbody>
</table>

4.5 Conclusions:

In summary, a novel series of 2-(4-(2-phenylimidazo[1,2-a]pyridin-6-yl)phenyl)-1H-benzo[d]imidazole (III a to III e) were synthesized. Synthetic methodology involved three steps. The obtained imidazole derivatives were characterized by mass, IR and NMR spectroscopic studies. Among the five different benzimidazole derivatives, III a and III d have been found to exhibit maximum activity, while other derivatives exhibited marginal activity with reference to CIPROFLOXACIN and GENTAMICIN.

References:

44. She, J.; Jiang, Z.; Wang, Y. Synlett 2009, 2023-2027.

97
Chapter - IV


\(^1\)H NMR spectrum of 4-(2-phenylimidazo[1,2-a]pyridin-6-yl)benzaldehyde
Mass spectrum of 4-(2-phenylimidazo[1,2-a]pyridin-6-yl)benzaldehyde
$^1$H NMR spectrum of 6-(2-methoxyphenyl)-2-phenylimidazo[1,2-a]pyridine
Mass spectrum of 6-(2-methoxyphenyl)-2-phenylimidazo[1,2-a]pyridine
Mass report of 2,6-diphenylimidazo[1,2-a]pyridine
$^1$H NMR spectrum of 6-(2-methoxyphenyl)-2-phenylimidazo[1,2-a]pyridine
Mass spectrum of 2-phenyl-6-(o-toly)imidazo[1,2-a]pyridine
$^1$H NMR spectrum of 6-(4-fluorophenyl)-2-phenylimidazo[1,2-a]pyridine
Mass spectrum of 6-(4-fluorophenyl)-2-phenylimidazo[1,2-a]pyridine
1H NMR spectrum of 6-(2-fluoro-4-methoxyphenyl)-2-phenylimidazo[1,2-a]pyridine
Mass spectrum of 6-(2-fluoro-4-methoxyphenyl)-2-phenylimidazo[1,2-a]pyridine
Mass spectrum of 6-(4-fluorophenyl)-2-phenylimidazo[1,2-a]pyridine
$^1$H NMR Spectrum of 2-(4-(2-phenylimidazo[1,2-a]pyridin-6-yl)phenyl)-1H-benzo[d]imidazole
Mass spectrum of 2-((4-(2-phenylimidazo[1,2-a]pyridin-6-yl)phenyl)-1H-benzo[d]imidazole
IR Spectrum of 2-(4-(2(phenylimidazo[1,2-a]pyridin-6-yl)phenyl)-1H-benzo[d]imidazole
Mass spectrum of 5-nitro-2-(4-(2-phenylimidazo[1,2-a]pyridin-6-yl)phenyl)-1H-benzo[d]imidazole
IR Spectrum of 4-nitro-2-(4-2(phenylimdazo[1,2-a]pyridin-6-yl]phenyl)-1H-benzo[d]imidazole
Mass spectrum of 5-chloro-2-((2-phenylimidazo[1,2-alpyridin-6-yl)phenyl]-1H-benzo[d]imidazole
1H NMR Spectrum of 6-chloro-2-(4-(2-phenylimidazo[1,2-a]pyridin-6-yl)phenyl)-1H-benzo[d]imidazole
IR Spectrum of 6-chloro-2-(4-2(phenylimdazo[1,2-a]pyridin-6-yl]phenyl)-1H-benzo[d]imidazole
1H NMR Spectrum of 6-bromo-2-(4-(2-phenylimidazo[1,2-a]pyridin-6-yl)phenyl)-1H-benzo[d]imidazole
Mass spectrum of 5-bromo-2-(4-(2-phenylimidazo[1,2-a]pyridin-6-yl)phenyl)-1H-benzo[d]imidazole
IR Spectrum of 5-bromo-2-(4-2(phenylimdazo[1,2-a]pyridin-6-yl)phenyl)-1H-benzo[d]imidazole
Mass spectrum of 5,6-dichloro-2-(4-(2-phenylimidazo[1,2-a]pyridin-6-yl)phenyl)-1H-benzo[d]imidazole
IR Spectrum of 5,6-dichloro-2-(4-2(phenylimdazo[1,2-a]pyridin-6-yl]phenyl)-1H-benzo[d]imidazole