A review of pyrazole and its derivative

Bhawna Poudyal and Bharghav G

Abstract
Pyrazole is a 2-neighbour nitrogen containing 5-membered heterocyclic organic compound with three carbon atoms. Pyrazole commonly known as 1,2 diazoles has been very popular now a days due to manifold uses. There are number of pyrazole derivative which has broad spectrum of biological activities like anti-bacterial, anti-microbial, anti-inflammatory, anti-convulsant, analgesic, anti-diabetic, anti-rheumatic, anti-cancer, and anti- tuberculosis. There are several applications of pyrazole core based organic molecules in various areas including pharmacy and agro-chemical industries. There is various method used for manufacture of pyrazole and its derivative. The purpose of the review was to collect various pharmacological actions which was reported in recent years made on its moiety. Pyrazole and its derivatives are prepared by dehydrogenating 2-pyrazoline or its derivative by process in which the reaction is carried out using sulfuric acid in the presence of iodine or of an iodine compound. Taking into considerations diversity in the biological activity, this nucleus has attracted attention of many researchers to study its skeleton chemically and biologically. This review highlights the different synthesis methods and the pharmacological properties of pyrazole derivatives. Development of pyrazole and its derivative has been reported by many scientists in decades.

Keywords: Pyrazole, Its derivative, 1, 2 Diazole

Introduction
Pyrazole may be any class of organic compound having heterocyclic ring composed of carbon atom an nitrogen atom in adjacent position of its structure. The simplest member of pyrazole family is pyrazole itself, a compound with molecular formulae C₃H₄N₂. The chemical reactivity of the pyrazole molecule can be explained by the effect of individual atoms. The N-atom at position 2 with two electrons is basic and therefore reacts with electrophiles. The N-atom at position 1 is unreactive, but loses its proton in the presence of base. The combined two N-atoms reduce the charge density at C3 and C5, making C4 available for electrophilic attack. Deprotonation at C3can occur in the presence of strong base, leading to ring opening. Protonation of pyrazoles leads to parazonium cations that are less likely to undergo electrophilic attack at C4, but attack at C3 is facilitated. The pyrazole anion is much less reactive toward nucleophiles, but the reactivity to electrophiles is increased. The pyrazole compounds are not known to occur in nature; they are usually prepared by reacting hydrazine’s with 1,3 -diketones. Many synthetic pyrazole compounds are of importance as dyes a medicine. Among them are antipyrine used as analgesic febrifuge; tartrazine most commonly used as yellow dye for food; phenylbutazone an ant inflammatory drug used in treatment of arthritis and series of dyes used as sensitizing agents in colour photography. Pyrazole derivatives play an important role in antitumor agents because of their good inhibitory activity against BRAF (V600E), GFR, telomerase, ROS Receptor Tyrosine kinase and Aurora-A kinase. In the addition, pyrazole derivatives also show good anti-inflammatory and anti-bacterial activities. Pyrazole are five membered heterocyle that constitute a class of compounds particularly useful in organic synthesis. They are one of the most studied groups of compounds among the azole family. Indeed, a huge variety of synthesis methods and synthetic analogues have been reported over the years. The presence of pyrazole nucleus in different structures leads to diversified applications in different structures leads to diversified areas such as technology, medicine and agriculture. In particular, they are described as inhibitors of protein glycation, antibacterial, antifungal, anti-cancer, antidepressant, anti-inflammatory, anti-tuberculosis, antioxidant as well as antiviral agents. Pyrazole or isoxazole derivatives are prepared by a palladium- catalysed four- component coupling of terminal alkaline, hydrazine (hydroxylamine), carbon monoxide under ambient pressure, and an aryl iodide.
Pyrazole are well known examples of aromatic heterocycles contain two nitrogen atoms in their five-membered rings. Pyrazole derivatives represent one of the most active classes of compounds and possess a wide spectrum of biological activities. Pyrazole derivatives are the subject of many research studies due to their widespread potential biological activities such as analgesic, antimicrobial and more. Diary pyrazole derivatives have been reported as nonnucleoside HIV-1 reverse transcriptase inhibitory activity. Kees et al. have described (4-substituted benzyl) (trifluoromethyl) pyrazole and pyrazolines which represent a new class of antidiabetic compounds. Cottineau et al. have reported the pharmacophore 3- methoxy- 1H- pyrazole-4-carboxlylic acid ethyl ester as hypoglycaemic agent. Das et al. have synthesised substituted pyrazole-3-one derivatives as potential hypoglycaemic agent.

Table 1: Pyrazole derivatives having pharmacological activities

<table>
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<tr>
<th>Drug</th>
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<td>Apixiban</td>
<td>Anticoagulant</td>
<td><img src="image6" alt="Apixiban Structure" /></td>
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</table>
Physical Properties of Pyrazole

Pyrazole has a five-membered aromatic ring structure consisting of two atoms of vicinal nitrogen, acidic pyrrole-like nitrogen with a single pair of aromatic electrons, simple sp²-hybridized nitrogen-like pyridine and three atoms of carbon, 7 and these combined features must be carefully taken into account in the context of reactivity. In the first instance, N-unsubstituted pyrazoles possess amphoteric properties, acting as both acids and bases, considering the presence of nitrogen. While the proton is easily donated by the acidic pyrrole-like NH group, the simple pyridine-like nitrogen can accept protons even more readily, and thus the basic character is typically prevalent. Nevertheless, substitutions on the ring can modulate these properties, as, for instance, electron donating groups were shown to increase the acidity of the pyrrole-like -NH group.9-10 In addition to the previous, the combination of two dissimilar and adjacent nitrogen atoms in this azole allows it to simultaneously donate and accept hydrogen bonds, which favours the establishment of intermolecular interactions, either among pyrazole molecules themselves and the nature of the substituents in the ring or between pyrazoles and neighbouring molecules that participate in proton transfer processes.

- Pyrazole is a colourless solid.
- With boiling point of 186-188°C,
- Melting point of 67-70°C,
- Pyrazole is having Pk_b = 11.5.
- Molecular weight 68.0776g/mol.
- Pyrazole is usually soluble in water

<table>
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<tr>
<th>Structure</th>
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<th>2d</th>
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Chemistry of Pyrazole

The high melting points and boiling point of pyrazole with 1-alkyl or 1-aryl substituent are due to the intermolecular hydrogen bonding which forms dimer molecules. It is tautomeric in nature (each of two molecules or isomers of compound which exist together in equilibrium, and are ready to get interchanged by migration of one or two atoms present within the molecules. pyrazole is weak In nature and forms organic salts with inorganic acids. The imino hydrogen group may be replaced by acyl group. Therefore, important properties of these molecules were analysed by comparing with the properties of benzene derivatives [3]. Like other nitrogen involving heterocycles, different tautomeric structures can be written for pyrazoles, unsubstituted pyrazole can be represented in three tautomeric forms [4].

The chemical properties of pyrazole can be explained by effect of individual atoms. the n- atom at position 2 with two electrons and therefore reacts with electrophile. the n- atom at position 1 is unreactive and loses its proton in the presence of base. The combined two n -atoms reduce the charge density at c3 and c5, making c4 available for electrophilic attack. Protonation of pyrazoles leads to parazonium cations and that less goes electrophilic attack at position of c-4, but the attack at c-3 is more facilitated the pyrazole for more effective nucleophiles.

Some of the important chemical properties of pyrazole is as follows

Basic Character

Pyrazole is a weakly basic compound and form pyrazole hydrochloride salts with inorganic acids.

Resonating Structure of Pyrazole

Pyrazole resist oxidation and reduction reaction due to loss of aromaticity, but may be hydrogenate catalytically, first to pyrazoline, and then to pyrazolidine. Both of these compounds are stronger bases than pyrazole.

Oxidation

Pyrazole ring system is resistant to oxidation but the side chain may be oxidized to carboxylic acid group in the presence of potassium permanganate. REDUCTION -Pyrazole ring can be reduced with molecular hydrogen and metal catalyst to pyrazole and pyrazolidine both are stronger bases than pyrazole.
Alkylation an Acylation
The free N-H group in pyrazole can be alkylated with alkylating agents such as alkyl halides, diazomethane an dimethyl sulphate or acylated using chloride and acetic anhydride.

Electrophillic Substitution Reaction
Pyrazole is an aromatic compound that exhibit all the properties of aromatic compound such as electrophilic substitution reaction e.g. halogenation, nitration sulfonation etc. In neutral or in basic medium, but not in acidic medium. The substitution occurs at C2-position through the formation of arenium ion intermediate.

Halogeneration
Halogeneration of pyrazole gives 4-mono halo pyrazoles e.g., 4-chloro, 4-iodo or 4-bromo pyrazole under controlled conditions but poor yields are obtained on the reaction of is thiazole and isoxazole. Bromine will attack at C–4, but with activating groups, present halogenation proceeds better. 3,4,5-tribromo pyrazole is formed efficiently in an alkaline solution; presumably, the pyrazole anion is the reacting species.

Nitration
Pyrazole undergo straight nitration at C-4, it gives 1-nitropyrazole but this can be rearranged to 4-nitropyrazole in acid at low temperature.

Sulphonation
Pyrazole reacts with fuming sulphuric acid to yield pyrazole 4–sulphonic acid.

Reactions of Pyrazoles with Nucleophiles
The presence of strong electron-withdrawing group on pyrazole assist nucleophile substitution.

Synthesis of Pyrazole \(^{[1-8]}\)
Pyrazoles are synthesized by the reaction of \(\alpha,\beta\)-unsaturated aldehydes with hydrazine and subsequent dehydrogenation:

\[
\begin{align*}
\text{O} + \text{H}_2\text{N}-\text{NH}_2 & \rightarrow \text{N} \\
\end{align*}
\]

Substituted pyrazoles are prepared by condensation of 1, 3-diketones with hydrazine (Knorr-type reactions). The Knorr \(^{[1]}\) pyrazole synthesis is an organic reaction used to convert a hydrazine or its derivatives and a 1,3-dicarbonyl compound to a pyrazole using an acid catalyst. The mechanism begins with acid catalysed imine formation, where the case of hydrazine derivatives the attack can happen on either carbonyl carbon and result in two possible products. For example, acetylacetone and hydrazine gives 3,5-dimethylpyrazole.

From Diketones
From 1,3 diketones the cyclocondensation of 1,3 – dicarbon compounds with the hydrazine derivatives is a simple and rapid approach to obtain polysubstituted pyrazoles. The first synthesis of the substituted pyrazoles was carried out in 1883 by Knorr who reacted beta diketone with hydrazine an give Regio isomers 2 and 3.

Scheme 1

\[
\text{CH}_3\text{C(O)CH}_2\text{C(O)CH}_3 + \text{N}_2\text{H}_4 \rightarrow (\text{CH}_3)_2\text{C}_3\text{HN}_2\text{H} + 2 \text{H}_2\text{O}
\]

Scheme 2

Synthesis of 1, 3 Substituted Pyrazole
An iron catalysed route for region selective synthesis of 1, 3 substituted pyrazoles from the reaction of diarylhydrazones and vicinal diol.

Scheme 3

Synthesis of Tri and Tetra Substituted Pyrazoles
A ruthenium (ii) – catalysed intramolecular oxidative CN coupling method for the facile synthesis of tri- an tetra-substituted pyrazoles. Dioxygen gas is employed as oxidant in this transformation and the reaction demonstrates excellent reactivity, functional group tolerance, and high yields.

Scheme 4

Synthesis of 3, 5 Substituted 1H-Pyrazole Synthesis-
A novel approach to the synthesis of pyrazole derivatives from tosylhydrazones of \(\alpha,\beta\)-unsaturated carbonyl compounds possessing a \(\beta\)-hydrogen is proposed, exploiting microwave activation coupled with solvent free reaction conditions.
Pyrazole derivatives (4a–4g) were synthesized in two steps
Firstly, hydrazone compounds (3a–3g) were generated by reacting compounds of acetophenone derivatives with different phenyl hydrazine derivatives in ethanol (EtOH) using acetic acid glacial as an acid catalyst. After that, hydrazones were reacted with Vilsmeier–Haack reagent which was produced by reacting dimethylformamidine (DMF) with phosphorus oxychloride (POCl₃) [40]. Different pyrazoles were produced as shown in Scheme. All synthesized pyrazoles were purified by flash chromatography. The structures of these compounds were confirmed by high-resolution mass spectrometry (HRMS), and ¹H NMR, ¹³C NMR spectral data and % purity of the synthesized compounds was determined by HPLC.

From 1,3-Diketones The cyclocondensation of the 1,3-dicarbonyl compounds with the hydrazide derivatives is a simple and rapid approach to obtain polysubstituted pyrazoles. The first synthesis of the substituted pyrazoles was carried out in 1883 by Knorr et al. who reacted β-diketone 1 with hydrazide derivatives to give two Regio isomers 2 and 3 (Scheme 1). Scheme 1.

Synthesis of polysubstituted pyrazoles form 1,3-dicarbonyl compounds. Girish et al. [12] described an efficient nano-ZnO catalysed green protocol for the synthesis of 1,3,5-substituted pyrazoles derivatives 6 by condensation of phenylhydrazine 5 with ethyl acetoacetate (Scheme 2). The main advantage of this protocol is the excellent yield (95%) achieved, short reaction time and easy work-up procedure.

General Synthesis-
Pyrazole and their derivatives were synthesized from hydrazine or its derivative and 1, 3 – dicarbon compound using an acid catalyst, the reaction is also known Knorr pyrazole synthesis.
Sucrow reported the synthesis of pyrazole using monomethyl hydrazones of dialkylaloacetates.
Hart an Brew have described the cyclization of 1, 3- bis (diazopropane) to pyrazole by concerted intermolecular 1,3-dipolar cycloaddition reaction.

Literature Review of Pyrazole and Its Derivatives
Eman M Flefel et al. [8] have reported new substitutes pyrazole thiazole and 1,2,4-triazole derivatives. The sugar hydrazones acetylated derivatives as well as their derived acrylic C-nucleoside analogues, and the thioglycosides of 1,2,4 -triazole derivatives were also prepared. The antitumor activity of some of the compounds showed significant activities.

Mohamedsalahk Youssef et al. [9] Have synthesized Ethyl 7-amino-3-(3-methyl-5-oxo-1-phenyl-2-pyrazolin-4-yl)-5-aryl-5H-thiazolo [3,2-a] pyrimidine-6-carboxylate was synthesized by the reaction of 4-(2-aminothiazol-4-yl)-3-methyl-5-oxo-1-phenyl-2-pyrazoline with arylidene ethyl cyanoacetate and it transformed to related fused heterocyclic systems via reaction with various reagents.

Rao Jyothi et al. [10] synthesized a novel series of 1,3 – trisubstituted pyrazoles by cyclo condensation reaction of chalcones and substituted hydrazides by irradiation under microwave energy and also by conventional. Compound 3g showed good activity against E. coli and Aeruginosa. Compound 3j showed good activity against the fungus A. fumigatus.

Sagar K. Mishra et al. [11] have reported the synthesis of a series of 1- (2, 4-dinitrophenyl)-3-(3-nitrophenyl)-5-(4-substituted phenyl)-2-pyrazolin-4-ones by the oxidation of 1-(2, 4-dinitrophenyl)-3-(3-nitrophenyl)-5-(4-substituted phenyl)- 4-bromo-2-pyrazolines with dimethyl-sulfoxide and assayed for in vitro antimicrobial activity. Most of the synthesized compounds did not exhibit significant inhibitory activity against the tested strains.
Satheesha Rai N [12] and Balakrishna Kalluraya et al. [12], have reported novel series of nitrofurran containing 1,3,4,5 tetra substituted pyrazole derivatives. Compound 3b showed highest anti-bacterial and antifungal activity than all other compounds.

Sahu SK et al., [13] have synthesized a series of novel 4-(5-substituted aryl-4, 5-dihydropyrazole-3-yl-amino) phenols by treating substituted aryl-N-chalcone amino phenols with hydrazine hydrate. It was observed increase in analgesic, anti-inflammatory and anti-microbial activities are attributed to the presence of 4-NO2, 2-OH and 4-Cl in phenyl ring at 5-position of pyrazoline ring of synthesized compounds.

Arcade ND et al., [14] have reported the conventional microwave assistance Synthesis of pyrazole containing 2, 4-disubstituted oxazol-5-one as a new class of antimicrobial agents. Compared to the conventional method, the microwave-assisted synthesis demonstrates several advantages, in terms of reaction time and overall yield. Compounds with electron withdrawing groups showed good antibacterial and antifungal activities. Among the compound tested, compound (3d) showed highest activity.

Biological Activities of Pyrazole an Its Derivative Anti-Inflammatory Activity
Inflammation is a multi-stage process that in critical step is supposed to be powered acutely released arachidonic acid and its prostaglandin like metabolites. NSAIDS (non-steroidal ant inflammatory drugs) is used for counteracting the pain of inflammation by counteracting cyclooxygenase enzyme. Some common examples of NSAIDs is aspirin, ibuprofen and naproxen. Carbaldehydeys were prepared and tested ant inflammatory an analgesics activity. Among the prepared compound exhibit maximum ant inflammatory activity. Novel series of pyrazole derivatives were reported by Tewari et al. (2014) [15] and evaluated in vivo ant inflammatory activity.

Ant inflammatory activity Berkhit et al. (2008) [15] reported a series of thiazolyl an thiadiazol derivatives of pyrazole to have an inflammatory activity. They observed the compounds were more active compound than the standard indomethacin and were found to be selective towards cox-2 enzyme. A series of compound was synthesized by Balbi et al. (2007) [15] (2,6,6- trimethyl -2-cyclohexin-1-ethyl -1-h pyrazole) were found to be potent inhibitors of neutrophil chemotactic responsiveness which could be considered as lead compounds and compared to standard diclofenac.

Anticancer Activity
Different pyrazole derivative is linked with pyrimidine, carbohydrazide, as well as ferrocenyl molecule with pyrazole which is effective with carcinoma of lung cell. Ohki et al. (2002) [20] synthesized the pyrimidinyl pyrazole derivative 1-(3,5, difluoro phenyl)-N-(E)-3-(1-pyrimidin-2-y1)-1H-pyrazol-4- yl) pieridine 4- amine as a new scaffold on antitumor agent an also having antiproliferative against lung cancer h cell lines and inhibited tubulin polymerization WEI et al. (2006) [21] reported a series of small molecules of compound ethyl 1- (20- hydroxy 30-aroxypropyl) -3- aryl-1h- pyrazole -5-carboxylatedervative which have the potency of supressing cancerous cell. Xia et al. (2007) [22] recorded a series of novel 1-arylmethyl -3-aryl1H pyrazole-5-carbohydraze derivatives which had inhibitory effects on the growth of A549 cells and induced the cell apoptosis.

Fan et al. (2008) [23] reported a series of novel 1-(3-(4-chlorophenoxynyl) phenyl)- 3-(4-chlorophenyl)-1H-pyrazole-5-carbohydrazide which is inhibiting the growth of A549 cells.

Li et al. (2012) [24] developed a series of 1H-pyrazole-4-carboxamide derivatives and reported their potential antiproliferation activity and Aurora-A kinase inhibitory activity. Among the compounds, N-(4-ethoxyphenyl)-1,3-diphenyl1H-pyrazole-4-carboxamide possess the most potent biological activity against HCT116 and MCF-7 cell lines with IC50 value of 0.39 and 0.46 µM, respectively.

Anti-Tubercular Activity
Manetti et al. (2006) [25] developed new inhibitors of mycobacterium tuberculosis. The compound 1-chlorophenyl-5- hydroxy-3-methyl-1H-pyrazol-4-yl(phenyl) methanone was found to be the most active compound against mic value 25 µM/ mL.50 As per continuation of previous work turned towards the identification of antimycobacterial compound with innovative structure, the compound (1-(4-bromophenyl)-5-hydroxy3-methyl-1H-
pyrazol-4-yl)(4-chlorophenyl)methane one of pyrazole derivative were synthesized by castagnolo et al. (2008) an was assayed as inhibitor of m tuberculosis H37Rv. The pyrazole derivatives with the p-bromophenyl group at the N1 position was showed to be very active. Pathak et al. (2014) [30] synthesized the various substituted 1-(3, 5-diary-4,5-dihydro-1H-pyrazol-1- yl) ethenone derivatives for their in vitro anti-tubercular activity against M. tuberculosis H37Rv strain.

Antidiabetic Activity
Cottineau et al. (2002) [32] were reported and developed a new series of substituted pyrazole-4-carboxylic acid for their antidiabetic activity. The results indicated that the prepared compound 3-methoxy-1Hpyrazole-4-carboxylicacid emerges as best hypoglycaemic agent among the series. Sharon et al. (2005) [35] prepared a new series of 5-[(5-arly-1Hpyrazol-3-yl) methyl]-1H-tetrazoles and isolated them for their in vivo anti-hyperglycaemic activity. Out of screen compound demonstrated 24.6% of blood glucose lowering activity at 100 mg/kg.57 Humphries et al. (2009) [34] synthesized the series of novel 4-pyrazoly1-2- aminopyrimidines as inhibitors of c-Jun-N-terminal kinases. This study led to the identification of compound (1s,4s)-4-(4-(3-tetrahydro-2H-pyran-3-yl)-1H-pyrazol-4-yl) pyrimidin-2-ylamino) cyclohexanol which showed good selectivity across a panel of diverse protein and lipid.

Anti-Tubercular an analgesic property
S. K. Sahu and co-workers [31] prepared a variety of pyrazole analogues and evaluated them for their analgesic affinity. Compounds 127 and 128 demonstrated highly potent analgesic activity against herpes simplex virus type-1 (HSV-1) and hepatitis-A virus (HAV).

K.K. Sivakumar and co-workers [35] constructed a library of coumarin-mediated pyrazole analogues. All the compounds were investigated for anti-inflammatory and analgesic activities. Compound 129 was observed to have the most powerful analgesic activity. Dias and Salvado95 prepared and exposed 4- methylthiophen-phen-3-yl) (1H-pyrazol-1-yl) methanone 130, which has an excellent analgesic affinity (Figure 11). A variety of unique 1-(5-(pyridin-3-yl)-1,3,4-thiadiazol-2-yl)-1H-pyrazole-4-carbaldehyde derivatives 131 were disclosed by Prathapa et al. [35] Their anti-tubercular and anti-oxidant properties were also scrutinized. All the prepared derivatives revealed significant anti-tubercular affinity.

Conclusion
Pyrazole represents a major pharmacophore with various biological properties, and some pyrazole- containing derivatives have already been used for therapeutic purposes. This shows that pyrazole derivatives are pharmacologically very potent and, therefore their design and synthesis is the potential area of research. It has been noted so far that the structural modifications of the basic structure of pyrazole, have allowed the preparation of new derivatives with a broad spectrum of biological activity, with the most important structural variations concerning the substituent at the 1- position, the carbon at the 3-position and the substituents at the 5-position.

Previous studies have shown that the structural modification on the different positions of basic molecule allows for improving its pharmacological profile, giving it antimicrobial, anticonvulsant, analgesic, anti-inflammatory, anti-viral anti-malarial and anti-inflammatory properties. For the moment, researchers have been drawn to the design of more potent pyrazole derivatives having great diversity. The pyrazole compounds were designed to have antibiotic activity which used docking studies.

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