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Recent Advances in Ultrasound Assisted Synthesis of 2,4,5-Trisubstituted Imidazoles: A Comparative Study of Effective Catalytic Systems

Vighnesh Khanvilkar[1], Sreela Dasgupta[2]
[1,2] Jai Hind College Autonomous, 23-24 Backbay Reclamation, Churchgate, Mumbai - 400020, India

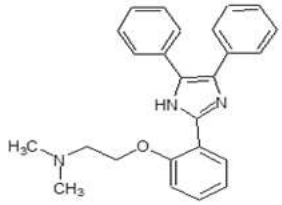
ABSTRACT: The imidazole moiety finds itself in a host of compounds having profound biological and medicinal properties. This has led to the quest to develop a cost effective, and desirably greener synthetic route to access this azole. In this review, ultrasonic irradiation assisted syntheses of trisubstitued imidazole derivatives have been discussed which make use of different environment friendly and efficient catalytic systems. They have been found to give excellent yield of product having high purity and with a shorter reaction time and has proved to be a viable alternative to traditional methods.

KEYWORDS: Catalysts, Green synthesis, Trisubstituted imidazole, Ultrasonication.

INTRODUCTION

In recent times, imidazole derivatives have gained increased significance owing to the fact that a large number of them have been found to be present in many diverse biologically and pharmacologically active products [1] such as Trifenagrel [2] , a platelet coagulation drug, Cyclooxygenase 2 (COX2) inhibitor [3], and many others. In particular, the 2,4,5 – trisubstituted imidazole moiety occurs in a number of herbicides [4], pesticides and fungicides [5]. Therefore , the syntheses of these imidazole derivatives have attracted much attention.

In recent years, an attempt to replace hazardous chemicals have seen the use of ultrasound irradiation, particularly in the synthesis of heterocycles. Numerous methods have also been reported where the catalytic activity in such syntheses has been enhanced using greener and non-toxic techniques [6].



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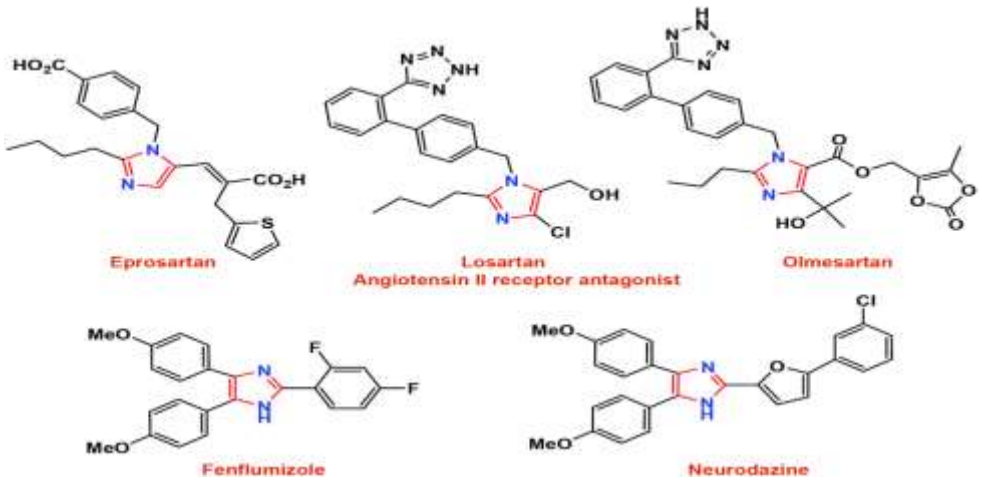

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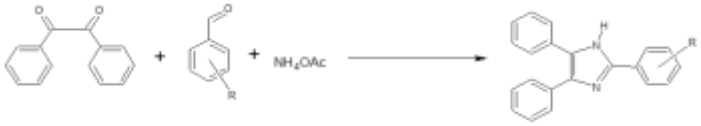
The first known synthesis of imidazole was reported by Radziszewski [7] and Japp [8] in which 2,4,5 –triphenyl imidazole derivatives were prepared using a 1,2 -dicarbonyl compound, ammonia, and aldehyde (Scheme 1). Subsequently, many other synthetic strategies have been developed over the years, for the synthesis of substituted imidazoles derivatives such as the Hetero-Cope rearrangement [9], four-component condensation of arylglyoxals, primary amines, carboxylic acids and isocyanides for Wangresin [10] and the three-component condensation of α-azido chalcones, aromatic aldehyde and substituted aniline for Erbiumtriflate [11]. However, many of these reactions were found to have certain disadvantages such as longer reaction time, low yield, tedious work up, or use of toxic reagents. This led to investigation for greener alternatives with a desirable enhanced reaction rate. Multicomponent reactions (MCR) or one-pot reactions in which three or more components react to give a single product such that almost all atoms of the starting material are incorporated into the product [12,13] qualify as green syntheses. This led to the exploration of the use of different catalysts in MCR for the synthesis of imidazole derivatives. Using the original scheme of Radziszewski and Japp, researchers started investigating the advantages of application of different catalysts such as uranyl nitrate hexahydrate supported on acidic alumina [14] or cellulosic sulfuric acid [15] both of which possess strong acidic properties, Y(TFA) [16], Molybdenum(IV) [17], Europium(OTf) [18], $SbCl_3/SiO_2$ [19], $ZrCl_4$ [20] , iodine [21], silica on sulfuric acid [22,23], L-proline [24], PEG-400 [25], NBS [26], Zr (acac) [27] and other fluoroboric acid derived catalysts [28]. However, all of these catalysts were found to have certain disadvantages such as use of very low pH, high cost factor, use of a large molar ratio of reagents or an increased reaction time. This led to the search for 'greener' alternatives which would correct all these deficiencies.

Ultrasound assisted reactions(UAS) have been found to decrease the reaction time to a great extent. Reactions which earlier took place in 24 hours could be completed within 2-3 hours using UAS. Ultrasound irradiation acts as an alternative energy source especially for such organic reactions which are difficult to conduct under thermal conditions. The salient features of Ultrasonic irradiation are enhanced reaction rates, formation of products of higher purity, easier work-up procedures, energy conservation and waste minimization [29,30]



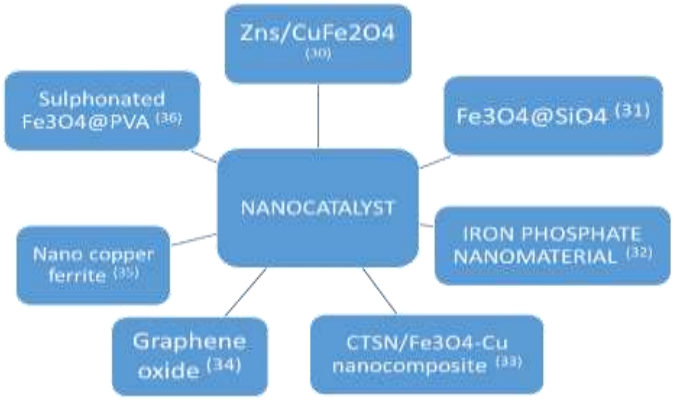
Scheme 1

In this review, we shall study the role of different catalytic reaction conditions under ultrasound irradiation in the synthesis of 2,4,5-trisubstituted imidazole derivatives.

RESULTS AND DISCUSSION

1. Nanocatalysts

In recent years, nanoscience has been an emerging field of interest for chemists because of their novel and unique applications. Scientists have studied the use of different nanocatalysts in the synthesis of diverse products. The advantages of using a nanocatalyst are good yields, less reaction time and recyclability. At times, many such catalysts could be easily recovered by a simple magnet and reused about 5-6 times. A few such nanocatalysts have been discussed below:



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Nanocatlysts used for the synthesis of imidazole derivatives

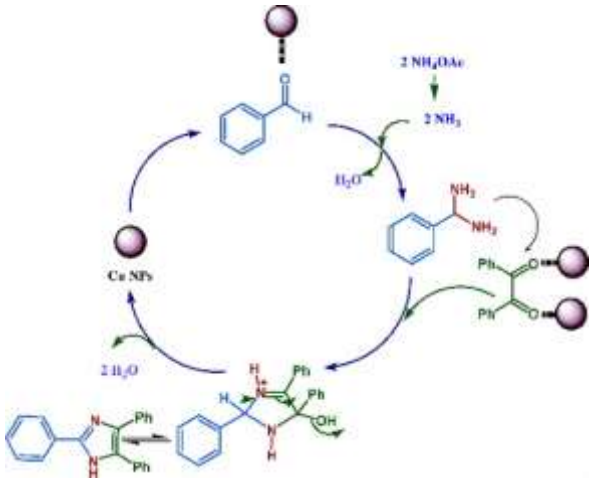
a. **Graphene oxide:**

The activity of graphene oxide was reported in 1962 by Hummers [31]. The GO is a two-dimensional catalyst with oxygen functional group having a large surface area (890 m^2 /g) which makes it a suitable candidate as a support catalyst [32]. It can be kept as a powder at room temperature and showed a good shelf life of a few months with no loss of activity.

Nano-catalyst	Solvent	Time (mins)		Yield (%)	
Graphene oxide		Ultasound present	Absent	Catalyst Present	Catalyst Absent
	H_2O	60	90	Trace	-
	THF	60	90	Trace	-
	$(CH_3CH_2)_2O$	60	90	Trace	-
	CH_2Cl_2	60	90	7	-
	$CHCl_3$	60	80	12	-
	None	15	60	95	30

b. CTSN/Fe_3O_4- Cu nanocomposite:

This nano catalyst is more specifically used in the synthesis of N-aryl imidazole derivatives [33]. The advantages of using this catalyst is that it is inexpensive, has a high magnetic character which helps separating it easily after completion of reaction without any requirement for complex purification methods. It can also be used a number of times without significant loss in its catalytic activity.



Nano-catalyst	Solvent	Time (mins) (UAS)	Yield (%)
CTSN/Fe_3O_4- Cu nanocomposite	DMF	10	88
	Ethyl alcohol	10	96
	H_20	10	98

Therefore, we can say that nanocatalysts have a synergistic effect with ultrasound irradiation which helps in reducing the reaction time to a great extent. Research is still going on to explore such advantages in other nanocatalysts.

2. Ionic Liquids

Ionic liquids have also been reported to catalyze organic reactions [34, 35]. In recent years, ionic liquids have been used multiple times in green synthesis [36]. Although ionic liquids were initially introduced as alternative green reaction media because of their

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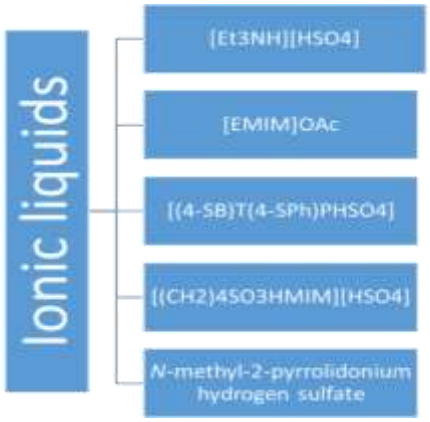
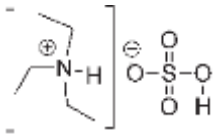
unique chemical and physical properties of nonvolatility, nonflammability, thermal stability, and controlled miscibility [37,38] ; nowadays they have evolved beyond this, showing their significant role in controlling reactions as catalysts [39, 40]. The use of ionic liquids as solvent and catalyst for chemical reactions offers a lot of advantages being environment friendly [41]. Also, it was observed that use of the ionic liquids as catalyst removes the use of external solvents. Solvent-free reaction has many advantages such as reduced pollution, cost-effective, efficient and simplicity in process and handling. These factors are especially very important in industry [42].

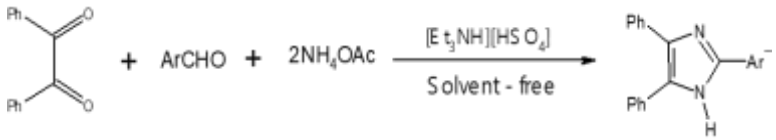
There are many ionic liquids which have catalyzed the synthesis of imidazole derivatives starting from benzil, aromatic aldehydes and ammonium acetate.



a. [Et_3NH] [HSO_4]



[Et_3NH][HSO_4]



Many ionic liquids have halogen atoms in them which may pose certain problems [43], under certain conditions. Therefore, there was a need to make halogen-free acids which could catalyze the reaction. Recently, halogen-free ionic liquids ([$(CH_2)_4SO_3HMIM$][HSO_4] [44] and N-methyl-2-pyrrolidonium hydrogen sulfate [45] have successfully catalyzed the synthesis of 2,4,5-trisubstituted imidazoles.

Entry	Temperature (ºC)	Catalyst(mol%)	Time (min)	Yield %
1.	100	10	70	77
2.	110	10	70	77
3.	120	10	55	89
4.	130	10	40	89
5.	140	10	40	90
6.	130	5	40	84

It was seen that, higher the reaction temperature, the more efficiently the reaction proceeded. The reaction was completed within 40 min to give the desired 2,4,5-triphenyl-1H-imidazole in 89 % yield at 130ºC in the presence of 10 mol % [Et_3NH][HSO_4]. A

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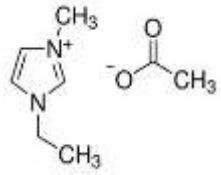
further increase in temperature did not however improve the yield. The optimum catalyst loading for $[Et_3NH][HSO_4]$ was found to be about 10 mol %. The catalytic activity of this has been compared with other catalysts later in this review.

b. [EMIM] OAc



An efficient and green method has been developed for the preparation of 2-aryl-4,5-diphenyl imidazoles catalyzed by 10 mol% of [EMIM]OAc under ultrasonic irradiation. The use of ultrasound combining ionic liquid in the three-component condensation brings up several advantages like less pollution, milder reaction conditions, good yields and simple work-up.

Catalyst	Amount of catalyst used (mol%)	Solvent	Method	Temp(°C)	Time (hr)	Yield
[EMIM]OAc	20	EtOH	ultrasound	25-30	1.5	94
	10	EtOH	ultrasound	25-30	1.5	96
	5	EtOH	ultrasound	25-30	1.5	77
	0	EtOH	ultrasound	25-30	1.5	15

Ionic liquids have therefore proved to be very useful in the synthesis of imidazole derivatives.

The table below gives a comparison between ionic liquids and other catalyst which have been used for the synthesis of 2,4,5 – trisubstituted imidazoles.

Entry	Catalyst	Reaction Condition	Time (mins)	Yield %	References
1.	Ammonium metavanadate	Reflux in EtOH	45	94	[46]
2.	Potassium aluminum sulfate	Ethanol, 70 C	150	93	[47]
3.	ZrOC128H2O	Ethanol–water (1:1, v/v)	30	92	[48]
4.	Polymer-supported zinc chloride	Reflux in EtOH	90	96	[49]
5.	Ceric ammonium nitrate	Ultrasonication, rt, ethanol– water (1:1, v/v)	100	98	[50]
6.	Tetrabutyl ammonium bromide	t-BuOH, 80 C	480	95	[51]
7.	PEG-400	PEG-400, 110 C	90	88	[52]
8.	[(CH2)4SO3HMIM][HSO4]	Solvent-free, 120^0 C	10	90	[53]
9.	N-methyl-2-pyrrolidonium hydrogen sulfate	Solvent-free, 100^O C	60	87	[54]
10.	[Et3NH][HSO4]	Solvent-free, 130^O C	40	89	[55]
11.	[EMIM]OAc	EtOH, 25°C	90	96	[56]

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3. Miscellaneous Catalysts

A few prominent miscellaneous catalysts which have been used for the synthesis of imidazole derivatives are:

1.Molecular iodine

2. 1,4-diazabicyclo[2.2.2]octane (DABCO)

3. L - Proline

4.Ytterbium triflate

a. Molecular iodine

In recent years, use of molecular iodine has found diverse uses [57] as it gives the product in excellent yields. It has many different advantages, being inexpensive, less toxic, and eco-friendly [58]. Different solvents were used to standardize the reaction conditions. Reaction goes well using I_2 / toluene, I_2 /acetonitrile, I_2 / dichloromethane and I_2 / ethanol. However, generally only ethanol is used as it is a relatively benign organic solvent. Moreover, ethanol involves only aqueous work up whereas with other solvents, hazardous solvents are required for the extraction of products.

Entry	Iodine mol %	Time (hrs)	Yield %
1.	0	4	0
2.	5	2.5	99
3	10	2	97
4	23	1.5	97
5.	30	1.5	99
6.	50	1	99

The data in the table shows that an increase in the quantity of iodine up to 50 mol% not only enhances the yield but also lessens the reaction time. However, results also clearly indicate that even 5 mol% of iodine is sufficient to catalyze the cyclo-condensation. No product was however reported even after 4 hours in the absence of iodine.

b. DABCO

DABCO has also been used as an efficient catalyst for the synthesis of imidazole derivatives [59]. The scheme of the reaction remains the same. When DABCO was used as a catalyst, the reaction proceeded smoothly at room temperature using ethanol as solvent, yielding the corresponding substituted imidazole in 85% yield after a reaction time of 24 hours. The same reaction when attempted in t-butanol at 60°–65°C, proceeded to completion within 12 h and yielded the corresponding imidazole in 92% yield [60].

ENTRY	SOLVENT	TIME (hrs)	YIELD(%)
1.	Methanol	12	67
2	Ethanol	12	80
3.	Iso-propanol	12	78
4.	Tert-butanol	12	92

c. L-Proline

In recent years, L-proline has been employed in different organic reactions due to its experimental simplicity, ease of handling, cost effectiveness and excellent solubility in water and organic solvents. L-Proline has been found to be a very efficient catalyst in different organic transformations [61- 64] and as an excellent promoter for copper-catalyzed reactions [65]. More recently, it has been used for the selective synthesis of 2-aryl-1-arylmethyl-1H-benzimidazoles from a wide range of substituted o-phenylenediamines and aldehydes [66].

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ENTRY	CATALYST(mol%)	SOLVENT	TEMP^{0}C	TIME (hrs)	YIELD %
1	-	MeOH	30	24	12
2	-	MeOH	60	24	25
3	5	MeOH	60	12	56
4	10	MeOH	60	12	78
5	15	MeOH	60	9	90
6	20	MeOH	60	12	88
7	15	EtOH	60	18	70
8	15	CHCl3	60	18	25
9	15	Toluene	60	24	30

CONCLUSION

This review summarizes the use of different catalysts in ultrasound assisted environment-friendly synthetic routes for the synthesis of 2,4,5- trisubstituted imidazole such that there is a significant increase in the reaction rates, selectivity, excellent yields and shorter reaction times. More research can be done in exploiting use of other catalysts which can be reused with comparable efficiency to give desired results.

Acknowledgement: The authors wish to thank Jai Hind College Autonomous for providing the necessary support in carrying out this review study.

List of Abbrevations used and their full forms:

PEG = Polyethylene Glycol
NBS = N-Bromo succimide
THF = tetrahydrofuran
GO = Graphene oxide
DABCO = 1,4- Diazabicyclo[2.2.2]octane
I2 = IODINE
EtOH = ethanol
MeOH = Methanol
DMF = DIMETHYLFORMAMIDE
EMIM(OAc) = 1- Ethyl-3-methylimidazolium Acetate
[Et3NH][HSO4] = Triethylammonium Hydrogen Sulphate

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