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PREPARATION OF 2,4,5-TRIARYLIMIDAZOLE DERIVATIVES USING GREEN *Syzygium Cumini* SEED CATALYST

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ABSTRACT
A simple, efficient, one-pot, and green synthesis of 2, 4, 5-triaryl imidazole was carried by three constituent reactions of benzil, ammonium acetate, and substituted aromatic aldehyde by using *Syzygium cumini* seed extract as a catalyst under reflux. The aqueous extract of *Syzygium cumini* seed as a biocatalyst is associated with high to excellent yield, green synthetic roughness, low reaction time, and eco-friendly operation. The catalyst contains different phytochemical contents, which are detrimental to improving the acidity of aqueous extract. The seed extract is non-hazardous, non-inflammable, low-cost, and environmentally green. All synthesized compounds were characterized by sophisticated spectroscopic techniques such as FT-IR, ^{1}H-NMR, ^{13}C-NMR, and Mass spectrometry.
Keywords: Green Synthesis, *Syzygium cumini,* Benzil, Substituted Aromatic Aldehyde, Triaryl Imidazole. Derivatives.

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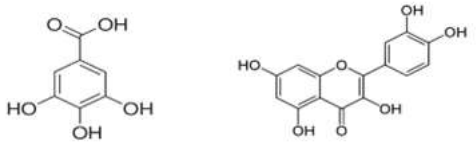
INTRODUCTION

Heterocyclic compounds are important candidates with diversified structural characteristics for a large number of biological, pharmaceutically active compounds, and agrochemicals. Imidazole is an important class of heterocyclic compounds owing to their prodigious biological activity and their utilization in synthetic, medicinal, and pharmaceutical chemistry. Imidazole is a five-membered aromatic heterocyclic compound containing two nitrogen atoms present in the 1, 3- position. The systematic name of this structure is 1, 3-diazole. It is highly a polar compound with high solubility in water. It is an amphoteric i.e. both acidic as well as basic in nature having basicity 100 times more than pyridine. Increased basicity results from resonance stabilization of the positive charge of the conjugate acid. Imidazole derivatives have great advantages for the synthesis of different kinds of Pharma compounds having a fine therapeutic utility such as anti-bacterial[1,2], cytotoxic[3,4], anti-inflammatory[5,6], anti-amoebic activity[7], anti-fungal[8,9], anti-HCV[10], anti-cancer[11], therapeutic activity[12], anti-tubercular[13], anti-convulsant[14], anti-candidal activity[15], anti-viral[16,17], Leishmanicidal[18], anti-proliferative[19,20], GABA uptake inhibitory[21], anti-tumor[22], and antimicrobial[23,24] activities, etc. The different substituted [1H] imidazole derivatives act as a Glucagon receptor[25], β-Raf Kinase[26], [p38α] MAP Kinase Inhibitors[27] and plant growth regulators[28], etc. In recent literature, numerous methods have been employed for the preparation of 2,4,5-arylimidazole derivatives using different types of catalysts such as $In(OTf)_3$ and $MgSO_4.7H_2O$[29], metal-organic framework $[Cu_2(BDC)_2(DABCO)]$[30], fly ash supported Bi_2O_3-ZnO[31], H_2SO_4-SiO_2[32], different nanoparticles such as nano copper ferrite[33], MgO nanoparticles[34], ZnO nanoparticles[35], Fe_3O_4, and Cu_2O nanoparticles[36], benzyl triphenyl phosphonium chloride (BTPPC)[37], ceric ammonium nitrate[38], HNO_3 in microwave irradiation[39], $ZrCl_4$[40], PEG-400[41], 3-picolinic acid[42], L-Proline[43], KMnO4/$CuSO_4$[44], etc. However, some of the recently reported synthetic methods for the preparation of imidazole derivatives suffer from various disadvantages such as the use of toxic, hazardous, or expensive acid catalysts, harmful reagents and solvents, and the prolonged period of time, poor product yield, and in cohesive reaction condition. Green chemistry is the modern concept of the removal, utilization, or development of non-hazardous compounds in the production and applications of chemicals, pharmaceuticals, and medicinal products. Bio-catalyzed synthetic reaction is the oldest chemical

modification known to humans with large importance in green Chemistry. *Syzygium cumini* seed is an important part of the plant. The chemical constituents of *Syzygium cumini* seed extract contain many anti-oxidant contents such as phenolic (Gallic acid), flavonoids (Quercetin), tannin (Gallic acid), and many natural products such as monoterpenoids.[45-47] Gallic acid is also called phenolic acid having 3, 4, 5-trihydroxy monocarboxylic acid as a functional group [P^H range-4.0][48], and quercetin is the most important part of flavonoid as an antioxidant. It is a part of the flavonols or group of flavonoids, related to a catechol moiety connected to the second position of the polyhydroxylated chromen-4-one core [P^H range-2.1].[49]



(a) (b)

Fig.-1: Structure of Gallic acid (a) and Quercetin (b) in Anti-Oxidant Content of Seed of *Syzygium cumini*

In the literature survey, seeds have been extracted with different solvents. Most commonly used solvents such as methanol, ethanol, water[50], etc. have been studied. Water is a common solvent used in the extraction of phytochemicals from the seed because of its ease of handling, non-poisonous, and environmentally safe nature. In the present study, we have carried out the synthesis of 2,4,5-triarylimidazole derivatives with benzil, ammonium acetate, and different substituted aromatic aldehydes using *Syzygium cumini* seed extract as a catalyst under reflux.

EXPERIMENTAL

Chemicals

Benzil, ammonium acetate, and aromatic aldehyde derivatives, all chemicals purchased were of AR grade and used without further purification.

General Procedure for the Preparation of Catalyst

10 g *Syzygium cumini* seed powder was taken in a clean 250 mL beaker then 100 mL water was added and boiled for 20 min. The prepared solution was filtered to remove unwanted impurities. The filtrate (aqueous extract) was used as a catalyst for the preparation of [1H] imidazole derivatives.

Synthesis of Substituted [1H] Imidazole Derivatives

In a clean 25 mL round bottom flask, a mixture of benzil (1 mmol), ammonium acetate (3 mmol), and substituted aromatic aldehydes (1 mmol) was taken and a catalytic amount of *Syzygium cumini* seed aqueous extract was added under refluxed condition. The progress of the reaction was monitored by thin-layer chromatography (TLC) on silica gel-coated Al-sheets by using a solvent mixture (n-hexane: Ethyl acetate-1:4). The crude imidazole product was filtered, washed, with water, and recrystallized by using ethanol.

Characterization

All the synthesized compounds were identified by comparing with a melting point, FT-IR, $^1H/^{13}C$-NMR, and Mass (LC-MS) data of known compounds. The FT-IR spectrum was recorded on SHIMADZU FT-IR 8400 using KBr pallets. The ^1H and ^{13}C-NMR spectra were recorded in CDCl3/DMSO-d6 on BRUCKER-400 MHz and the LC-Mass spectrum was recorded on SHIMADZU MODEL-8045 at the ESI-APCI interface.

Scheme-1

Spectral Analysis

1a: 2, 4, 5-Triphenyl-1H-Imidazole. White solid, M.P- 272-273 °C.

FT-IR (KBr, cm^{-1}): 3062.42, 1659.38, 1594.58, 1460.73, 1210.74, 1127.66, 765.48, 697.03, ^1H-NMR (400 MHz, DMSO-d6): δH 11.835 (s, 1H, NH), δH 8.008-7.215 (m, 15H, ArH), ^{13}C-NMR (DMSO-d6):- δC

194.63, δC 146.31, δC 134.95-125.55, (+)ESI-HRMS *m*/*z*: calculated for [$C_{21}H_{16}N_2$ + H^+] 297.0, observed 297.40

1b: 2-(4-Hydroxyphenyl)-4, 5-diphenyl-1H-Imidazole. White solid, M.P- 234-235 °C.
FT-IR (KBr, cm^{-1}): 2969.30, 2722.69, 1456.79, 1359.0, 1167.55, 973.34, 841.54, 808.68, ^{1}H-NMR (400 MHz, DMSO-d6): δH 9.366 (s, 1H, NH), δH 7.846 (d, 2H, ArH), δH 7.470 (d, 2H, ArH), δH 7.452-6.753 (m, 10H, ArH), ^{13}C-NMR (DMSO-d6):- δC 158.0, δC 146.67, δC 128.38-126.98, δC 115.58, (+)ESI-HRMS *m*/*z*: calculated for [$C_{21}H_{16}N_2O$ + H^+] 313.0, observed 312.40

1c: 2-(4-Chlorophenyl)-4, 5-diphenyl-1H-Imidazole. White solid, M.P- 258-260 °C.
FT-IR (KBr, cm^{-1}): 3522.84, 1695.80, 1591.12, 1486.05, 1328.11, 1186.37, 1089.69, 979.12, 823.67, 701.88, ^{1}H-NMR (400MHz, DMSO-d6): δH 9.366 (s, 1H, NH), δH 7.852 (d, 2H, ArH), δH 7.831 (d, 2H, ArH), δH 7.554-7.265 (m, 10H, ArH), C^{13}-NMR (DMSO-d6):- δC 145.04, δC 133.47, δC 129.34-126.95, (+)ESI-HRMS *m*/*z*: calculated for [$C_{21}H_{16}N_2Cl$ + H^+] 331.0, observed 330.80

RESULTS AND DISCUSSION

Synthesis of [1H] imidazole by using *Syzygium cumini* Seed Extract

The one-pot multicomponent reaction between benzil, ammonium acetate, and substituted aromatic aldehyde using *Syzygium cumini* seed extract as a catalyst under reflux to form various substituted [1H] imidazole was optimized with varying different parameters. The results obtained are shown in Table-1. Herein, different types of solvents were investigated at 80 °C with 5 % mol of catalyst. We have carried out reactions in different amounts of catalyst and the product yield was increased from 28 % to 96 % by increasing the quantity of catalyst. It was found that 15 % mol of catalyst is optimum. Further, increasing the amount of catalyst or temperature does not affect the percentage yield of the product as shown in Table-2. Under the optimized reaction condition, different substituted aromatic aldehydes were reacted with benzil and ammonium acetate to obtain substituted [1H] imidazole. In all the cases, reactions were completed in a reasonable time and substituted [1H] imidazole derivatives were formed in high product yield as shown in Table-3.

Table-1: Effect of Various Solvents for the Synthesis of 2, 4, 5-Trisubstituted [1H] Imidazole Derivatives

S. No.	% mol of catalyst	Solvent	Temp (°C)	Time (Min)	Yield (%)
1	5	Ethanol	80	60	42 %
2	5	Methanol	80	60	35 %
3	5	Toluene	80	60	34 %
4	5	Acetonitrile	80	60	40 %
5	5	Chloroform	80	60	38 %
6	5	DMSO	80	60	36 %
7	5	Dichloromethane	80	60	38 %
8	5	Solvent-free	80	60	30 %
9	5	Water	80	60	53 %

Table-2: Optimization of Catalyst for the Synthesis of 2, 4, 5-Trisubstituted [1H] Imidazole Derivatives

S. No.	% mol of Catalyst	Solvent	Temp (°C)	Time (Min)	Yield (%)
1	------	Water	80	60	30 %
2	5	Water	R.T.	60	28 %
3	5	Water	80	60	53 %
4	10	Water	80	60	78 %
5	15	Water	80	20	96 %
6	15	Water	100	20	96 %
7	20	Water	80	20	96 %
8	25	Water	80	20	96 %

Syzygium cumini seed extract acts as an acid catalyst for the synthesis of [1H] imidazole derivatives. This methodology facilitates the synthesis of a group of functionalized [1H] imidazole derivatives with electron-withdrawing and electron-donating groups. It was observed that electron-donating substituents of aromatic

aldehyde decrease the reaction rate with less yield of the product while electron-withdrawing substituents of aromatic aldehyde increase the rate of reaction with a higher yield of product.

Table-3: *Syzygium cumini* Seed Catalyzed by Synthesis of 2, 4, 5-trisubstituted [1H] Imidazole Derivatives

Entry	R-Ar-CHO	Product	Time (Min)	Yield (%)	M.P. (°C)
1a	H-Ar-CHO	2, 4, 5-triphenyl [1H] imidazole	20	96	271-272
1b	p-OH-Ar-CHO	2-(4-Hydroxyphenyl)- 4, 5-diphenyl [1H] imidazole	20	95	233-234
1c	p-Cl-Ar-CHO	2-(4-Chlorophenyl)- 4, 5-diphenyl [1H] imidazole	20	96	259-261
1d	p-OCH_3-Ar-CHO	2-(4-Methoxyphenyl)- 4, 5-diphenyl [1H] imidazole	20	94	227-229
1e	p- NO_2-Ar-CHO	2-(4-Nitrophenyl)- 4, 5-diphenyl [1H] imidazole	20	96	199-201
1f	2-OH-Ar-CHO	2-(2-Hydroxyphenyl)- 4, 5-diphenyl [1H] imidazole	20	95	210-211
1g	2-Cl-Ar-CHO	2-(2-Chlorophenyl)- 4, 5-diphenyl [1H] imidazole	20	96	193-194
1h	2-NO_2-Ar-CHO	2-(2-Nitrophenyl)- 4, 5-diphenyl [1H] imidazole	20	96	299-301
1i	Furfural	2-(Furfural-2-yl)- 4, 5-diphenyl [1H] imidazole	20	95	201-203
1j	p-Dimethyl amino	2-(4- Dimethyl amino phenyl)- 4, 5-diphenyl [1H] imidazole	20	95	257-258

CONCLUSION

In conclusion, we have developed a facile, efficient, one pot and green protocol for the synthesis of 2,4,5-trisubstituted [1H] imidazole derivatives from the three-component reaction of benzil, ammonium acetate, and substituted aromatic aldehyde using *Syzygium cumini* seed extract as a catalyst under reflux. The method has great benefits such as short reaction time, easy work-up process, and high yield. The *Syzygium cumini* seed catalyst is easy for separation, non-toxic, low waste, environmentally benign, and cost-effective, thereby satisfying maximum principles of green chemistry.

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