

Green Synthesis of 2-(Substituted Phenyl)-4,5-diphenyl-1H-imidazole Using Natural Heterogeneous LPP Catalyst

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Abstract: In this study, we describe the synthesis of substituted 2,4,5-triaryl imidazole through a one-pot, three-component reaction involving benzyl, substituted aldehyde, and ammonium acetate, using lemon peel powder (LPP) as a catalyst in ethanol as the solvent. A notable aspect of this approach is the catalytic efficiency of LPP. The findings demonstrate that LPP, under ethanol solvent conditions at 70°C, serves as an optimal catalyst for the formation of fused products, yielding excellent results. This method also proved effective on a gram scale, highlighting its potential for both industrial and academic applications, producing high-purity products with excellent yields.

Keywords: green synthesis; LPP catalyst; benzyl; substituted aldehyde.

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1. Introduction

Multicomponent reactions establish an emerging tool as this reaction plays an important role for developed and educational research groups due to their less reaction time as well as the inhibition of the separation of intermediates, purification of products, and environmentally friendly nature [1]. Imidazole was revealed in 1840 by the reaction of formaldehyde, glyoxal, and ammonia. Inventor Heinrich Debus was the first to present the synthesis of imidazole results [2,3]. Imidazole is an important heterocyclic compound that is present in the natural product and biological system, Figure 1 [4].

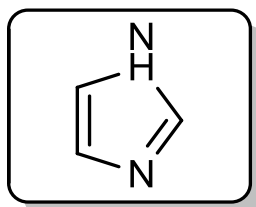


Figure 1. General structure of imidazole.

Imidazole is the most significant structure of some predictable components of the human organism, which comprise histamine, amino acid, biotin, purines, and the structure of DNA base construction [5]. Imidazole mostly involves five-membered rings containing two nitrogen atoms at 1,3 non-adjacent positions, which establish its acidity and basicity (Figure 1) [6]. Imidazole ring-containing compounds have an extensive range of biological and

pharmacological actions[7]. Imidazoles keep a widespread range of biological activities such as anti-cancer[8], anti-tubercular[9], anticoagulants[10], antiviral[11], analgesic[12], anti-inflammatory[13], antibacterial[14], antihypertensive[15]. Some important imidazole-containing drugs are shown in (Figure 2)[16].

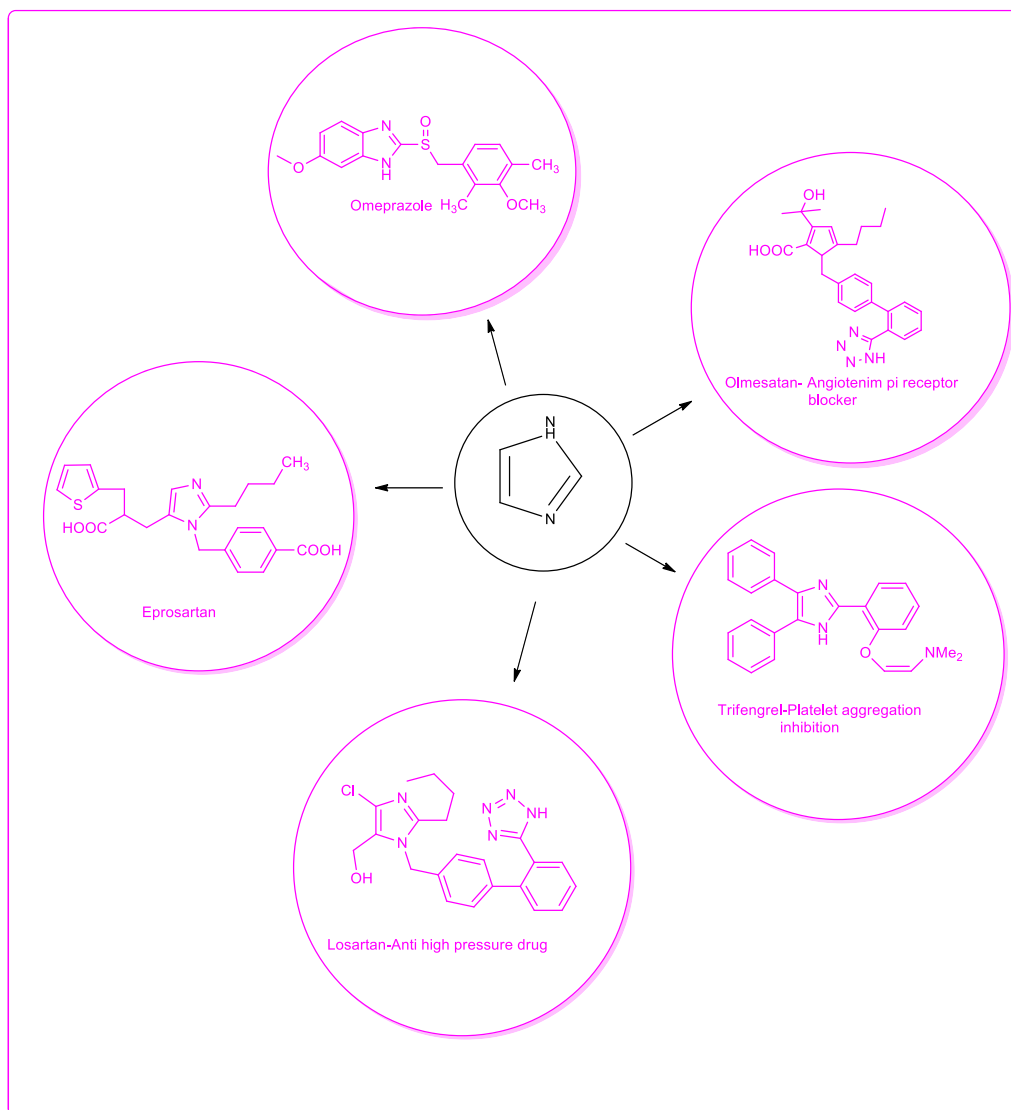


Figure 2.Imidazole-containing ring.

Lemon is a significant therapeutic plant that goes to the Rutaceae family. During the manufacture of citrus juice, a very great quantity of byproduct waste, such as peels, is formed every year, and also lemon peels show biological activity like anticancer, antibacterial, antiviral, and antifungal, etc., [17]. The acidic constituent called citric acid, besides carboxylic acid, is controlled in the chemical composition of lemon peel, other than water[18]. The majority of chemical compounds in lemon fruits are alkaloids[19]. Lemon peel controls crude fibers (15.18%), protein (9.42), and crude fat (4.98%). Ash pleased of lemon peel is 6.26%, and lemon juice is about 5% acid, which gives lemons a sour taste (pH 2-3)[20].

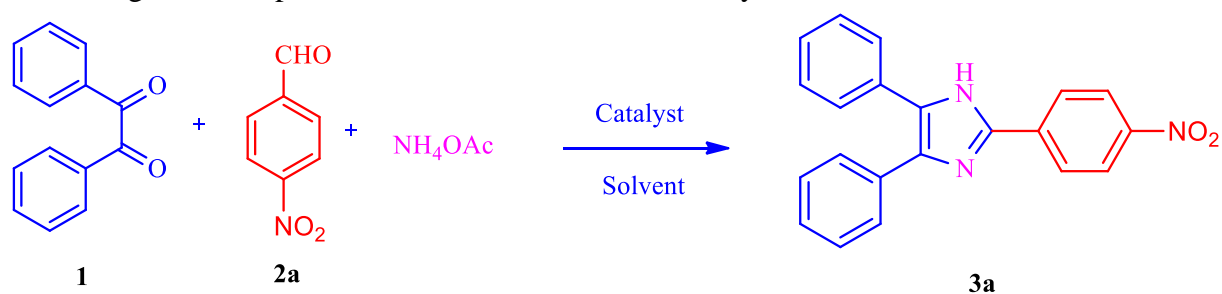
In our previous work, we concentrated on synthesizing novel hybrid molecules, driven by our commitment to designing and creating new compounds [21-31]. In our current work, we developed a green protocol for synthesizing 2,4,5-triarylimidazole using benzil, substituted aromatic aldehyde, and ammonium acetate, with lemon peel powder as a catalyst in ethanol as a green solvent at room temperature, achieving a short reaction time.

2. Materials and Methods

All the manufactured compounds were characterized with ^1H NMR, IR, and Mass spectra. All chemicals were spectrochem made and used without purification. ^1H NMR spectra were recorded on 500MHz in CDCl_3 solvent. Chemical shifts were in δ ppm. The reaction growth was monitored on a TLC plate with 30% ethyl acetate and 70% n-hexane as the mobile phase. Melting points recorded in capillaries open at one end were uncorrected.

3. Results and Discussion

We have synthesized substituted 2,4,5-triaryl imidazole through a one-pot, three-component reaction involving benzyl, substituted aldehyde, and ammonium acetate and screening of various catalysts (Scheme 1, Table 1). Initially, the model reaction was performed using $\text{InCl}_3 \cdot 3\text{H}_2\text{O}$, which yielded low conversion and required extended reaction times (Entry 1, Table 1). Subsequently, various coupling reagents, including L-Proline, K_2CO_3 , and KH_2PO_4 , were investigated (Entries 2–4, Table 1). These catalysts facilitated the reaction within 9, 7, and 1 hour, respectively. The optimal result was achieved using lemon peel powder (LPP) as a catalyst, producing product 3a with a 91% yield (Entry 5, Table 1). Among the tested reagents, LPP proved to be the most effective catalyst for the conversion.



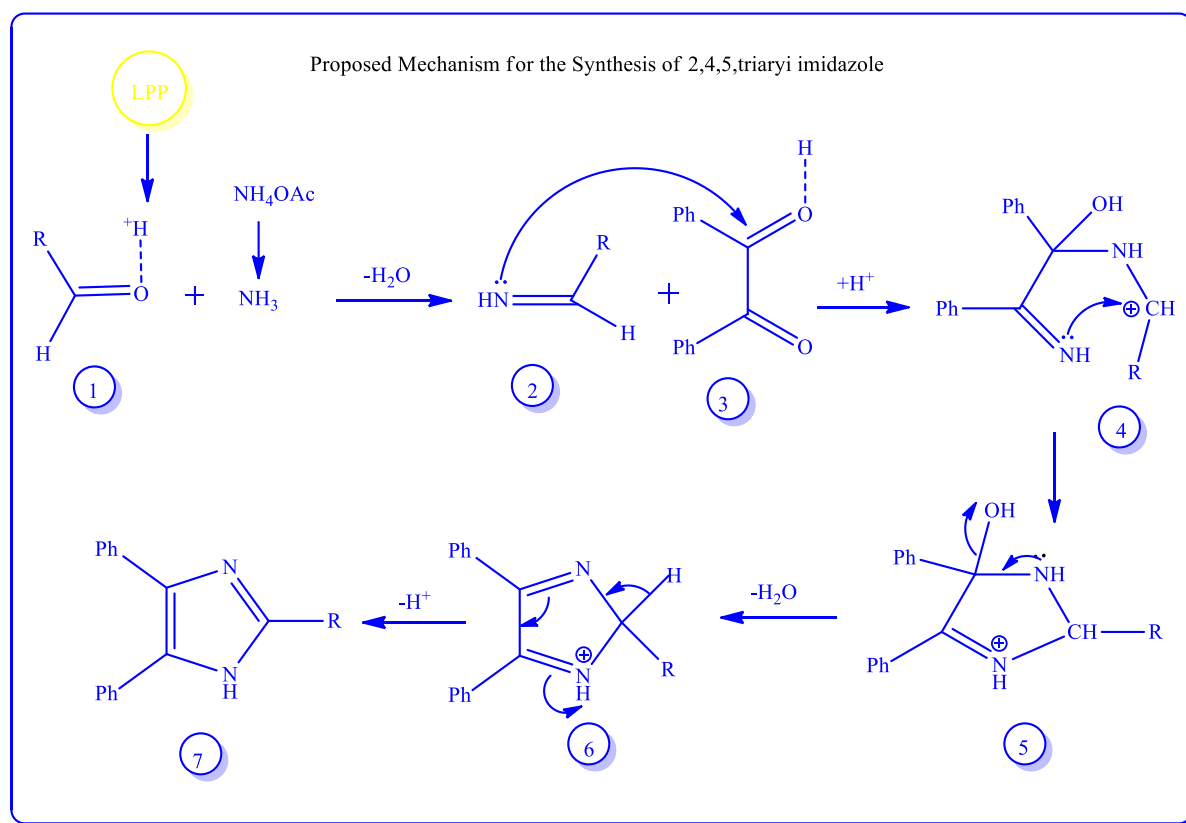
Scheme 1. Screening of solvent and catalyst of 2-(4-nitrophenyl)-4,5-diphenyl-1H-imidazole.

The proposed mechanism for synthesizing the imidazole molecule is illustrated in Scheme 2.

Table 1. Screening of catalyst on the model reaction.

| Entry | Catalyst | Temperature | Time (h) | Catalyst (wt. %) | Yield (%) |
|-------|---|------------------|----------|------------------|-----------|
| 1 | $\text{InCl}_3 \cdot 3\text{H}_2\text{O}$ | Reflux | 8 | 10 | 82 |
| 2 | L-Proline | Reflux | 9 | 10 | 87 |
| 3 | K_2CO_3 | Reflux | 7 | 10 | 85 |
| 4 | KH_2PO_4 | Reflux | 1 | 10 | 89 |
| 5 | LPP | Room temperature | 0.5 | 10 | 91 |

The reaction was then carried out in various solvents, with results summarized in Table 2. Under solvent-free conditions, the yield was low at 35% (Entry 2, Table 2). Moderate yields were obtained in water (45%, Entry 1, Table 2) and acetone (65%, Entry 3, Table 2). In contrast, ethanol, a green solvent, facilitated the completion of the reaction within 45 minutes, yielding an excellent 91% (Entry 4, Table 2). Consequently, ethanol was identified as the optimal solvent for this model reaction. Following this, catalyst concentrations were optimized using lemon peel powder (LPP) as a catalyst (Table 3). Concentrations of 5, 10, 15, and 20 wt.% were tested, with the highest yield (91%) achieved in 45 minutes using 10 mol.% catalyst. Further increases in catalyst concentration did not improve the yield.



Scheme 2. Proposed mechanism of synthesis of the imidazole molecule.

Table 2. Effect of solvent on the model reaction.

| Entry | Solvent | Time (min.) | Catalyst (wt. %) | Yield (%) |
|-------|---------|-------------|------------------|-----------|
| 1 | Water | 60 | 10 | 45 |
| 2 | - | 180 | 10 | 35 |
| 3 | Acetone | 120 | 10 | 65 |
| 4 | Ethanol | 30 | 10 | 91 |

Table 3. Effect of catalyst concentration on model reaction.

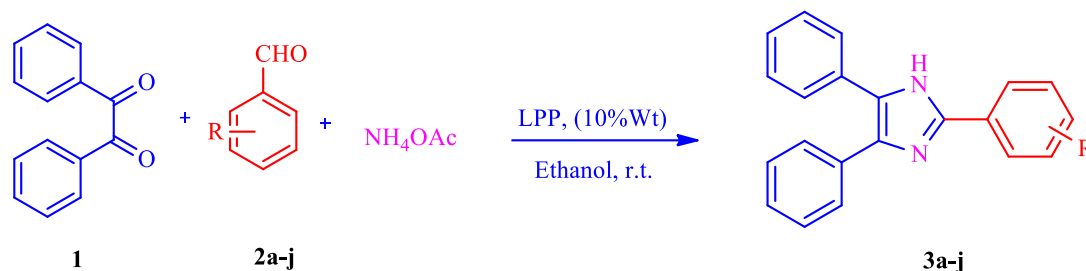
| Entry | Catalyst concentration (wt.%) | Yield (%) |
|-------|-------------------------------|-----------|
| 1 | 5 | 60 |
| 2 | 10 | 91 |
| 3 | 15 | 90 |
| 4 | 20 | 89 |

We have synthesized substituted 2,4,5-triaryl imidazole through a one-pot, three-component reaction involving benzyl, substituted aldehyde, and ammonium acetate and screening of various catalysts (Scheme 1, Table 1). Initially, the model reaction was performed using $\text{InCl}_3 \cdot 3\text{H}_2\text{O}$, which yielded low conversion and required extended reaction times (Entry 1, Table 1). Subsequently, various coupling reagents, including L-Proline, K_2CO_3 , and KH_2PO_4 , were investigated (Entries 2–4, Table 1). These catalysts facilitated the reaction within 9, 7, and 1 hour, respectively. The optimal result was achieved using lemon peel powder (LPP) as a catalyst, producing product 3a with a 91% yield (Entry 5, Table 1). Among the tested reagents, LPP proved to be the most effective catalyst for the conversion.

3.1. General procedure for the synthesis of substituted 2, 4, 5-triaryl imidazole (3a-j).

A mixture of Substituted benzaldehyde (1 mmol), benzil (1 mmol), ammonium acetate (2.5 mmol), and lemon peel powder (10wt.%) was stirred in 5ml of ethanol solvent under room

temperature condition (Scheme 3). Later, the reaction was accomplished as established by the TLC plate, and the mixture was poured onto crushed ice, stirred for 10 minutes, and filtered off. The solid product was purified by hot ethanol to obtain pure substituted imidazole products (**3a-j**). The physical data of synthesis of 2, 4, 5-triaryl imidazole derivative (**3a-j**) is summarised in Table 4.



Scheme 3. Synthesis of 2-(substituted phenyl)-4,5-diphenyl-1H-imidazole.

Table 4. Physical data of synthesis of 2, 4, 5-triaryl imidazole derivative (**3a-j**).

| Sr. No. | Aldehyde | Molecular formula | Time (Min.) | Yield% | M.P. (°C) observed | M.P. (°C) reported |
|---------|---|--|-------------|--------|--------------------|-------------------------|
| 3a | 4-NO ₂ C ₆ H ₄ - | C ₂₁ H ₁₅ N ₃ O ₂ | 60 | 81 | 231-233 | 232-233 ^[21] |
| 3b | 4-BrC ₆ H ₄ - | C ₂₁ H ₁₅ BrN ₂ | 45 | 90 | 244-246 | 245-246 ^[22] |
| 3c | 4-OHC ₆ H ₄ - | C ₂₁ H ₁₆ N ₂ O | 50 | 83 | 264-266 | 265-267 ^[23] |
| 3d | 2-ClC ₆ H ₄ - | C ₂₁ H ₁₅ ClN ₂ | 60 | 71 | 194-196 | 195-197 ^[21] |
| 3e | 2,4-Cl ₂ C ₆ H ₄ - | C ₂₁ H ₁₄ Cl ₂ N ₂ | 60 | 86 | 173-175 | 174-176 ^[24] |
| 3f | C ₆ H ₅ - | C ₂₁ H ₁₆ N ₂ | 45 | 91 | 272-274 | 273-275 ^[23] |
| 3g | 2-OCH ₃ C ₆ H ₄ - | C ₂₂ H ₁₈ N ₂ O | 60 | 80 | 211-213 | 212-213 ^[23] |
| 3h | 3-NO ₂ C ₆ H ₄ - | C ₂₁ H ₁₅ N ₃ O ₂ | 60 | 80 | 313-315 | 314-316 ^[16] |
| 3i | 4-ClC ₆ H ₄ - | C ₁₂ H ₁₅ ClN ₂ | 45 | 89 | 255-258 | 257-260 ^[16] |
| 3j | 2,6-Cl ₂ C ₆ H ₄ - | C ₂₁ H ₁₄ Cl ₂ N ₂ | 60 | 80 | 173-175 | 174-176 ^[14] |

3.1.1. 2-(4-nitrophenyl)-4,5-diphenyl-1H-imidazole.

Yield: 81%, M.P. 231-233°C, IR (KBr) Cm⁻¹: 1321.24 (-C-C), 1651(-C=N), 2964.59(-CH), 3323.35 (-NH), ¹H NMR Spectra: (500 MHz, CDCl₃) δ (ppm): 7.40-8.50 (m, 10H, Ar-H), 8.10 (d, 2H, Ar-H), 8.34 (d, 2H, Ar-H), 12.90 (s, br, NH). GC-MS: 342.47 (M+1)

3.1.2. 2-(4-bromophenyl)-4,5-diphenyl-1H-imidazole.

Yield: 90%, M.P. 244-246°C, IR (KBr)Cm⁻¹: 873.75 (C-C), 1658.78 (-C=N), 3066.82 (-CH), 3315.63 (-NH), ¹H NMR Spectra: (500 MHz, CDCl₃) δ (ppm): 7.49-7.53(m, 10H, Ar-H), 7.68(d, 2H, Ar-H), 7.70(d, 2H, Ar-H), 11.59 (s, br, NH), ESI-MS:376.00 (M+1)

3.1.3. 4-(4,5-diphenyl-1H-imidazole-2-yl) phenol.

Yield: 83%, M. P. 264-266°C, IR (KBr) Cm⁻¹: 1639.49 (C=C), 1689.64 (-C=N), 3350.35 (-OH), 3404.36 (-NH), ¹H NMR Spectra: (500, CDCl₃) δ (ppm): 5.37 (s, br, OH), 6.89 (d, 2H, Ar-H), 7.43-7.54 (m, 10H, Ar-H), 7.93 (d, 2H, Ar-H), 11.54 (s, br, NH), ESI-MS: 313.30 (M+1).

3.1.4. 2-(2-Chlorophenyl)-4,5-diphenyl-1H-imidazole.

Yield 71%, M.P.194-196°C, IR (KBr)Cm⁻¹: 1645.28 (-C=N), 3473.80 (-NH), ¹H NMR Spectra: (500 MHz, CDCl₃) δ (ppm): 7.40-7.49 (m, 10H Ar-H), 7.37(dd, 1H, Ar-H), 7.41(dd, 1H, Ar-H), 7.57 (d, 1H, Ar-H), 7.75 (d, 1H, Ar-H), 12.10 (s, br, NH), ESI-MS:331.91 (M+1).

3.1.5. 2-(2,4-dichlorophenyl)-4,5-diphenyl-1H-imidazole.

Yield: 86%, M. P. 173-175°C, IR (KBr) Cm^{-1} : 1645.28 (-C=N-), 3471.87 (-NH), ^1H NMR Spectra :(500 MHz, CDCl_3) δ (ppm): 7.42-7.52 (m, 10 H, Ar-H), 7.45 (d, 1H, Ar-H), 7.82(d, 1H, Ar-H), 7.77 (s, 1H, Ar-H), 11.82 (s, br, NH), ESI-MS: 365.00 (M+1).

3.1.6. 2,4,5-triphenyl-1H-imidazole.

Yield: 91%, M.P. 272-274°C, IR Spectra: IR (KBr) Cm^{-1} : (-C-C-) 1170.79, (-C=C) 1567.42, (-C=N) 1658.78, (-CH) 3066.82, (-NH) 3408.22, ^1H NMR Spectra: (500 MHz, CDCl_3) δ (ppm): 6.79-7.90 (m, 15H, Ph-H), 11.90 (s, br, NH), ESI-MS: 297.2 (M+1).

4. Conclusions

We explored the use of an LPP catalyst in a one-pot, three-component synthesis of 2,4,5-triaryl imidazoles at room temperature, employing ethanol as the solvent. This method proved effective for a variety of compounds, including both heterocyclic and aromatic aldehydes. The environmentally friendly reaction conditions offer benefits such as simple work-up, shorter reaction times, and improved yields. The catalyst serves as an economical alternative, being easy to prepare, biodegradable, recyclable, and not as harmful to the environment as conventional solvents. The products were obtained in high purity and yield within short reaction times. The methodology further supports green chemistry by allowing the recycling and reuse of LPP, demonstrating excellent biodegradability. Additionally, this approach showed promising results when scaled up to gram quantities, suggesting its potential for both industrial and academic applications.

Author Contributions

Conceptualization, methodology, S.D. P.K.; D.P.; R.P.; investigation, resources, data curation, writing—original draft preparation, S.D. P.K.; and D.P.; writing—review and editing, D.P.; visualization, S.D. P.K.; supervision, D.P.; project administration, D.P.; funding acquisition, D.P. All authors have read and agreed to the published version of the manuscript.

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Informed Consent Statement

Not applicable.

Data Availability Statement

Data supporting the findings of this study are available upon reasonable request from the corresponding author.

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Conflicts of Interest

The authors declare no conflict of interest.

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