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# "THREE-COMPONENT, ONE-POT SYNTHESIS OF 2,4,5-TRISUBSTITUTED IMIDAZOLE'S CATALYZED TAMARIND BY MICROWAVE IRRADIATION"

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## ABSTRACT

In this present study, tamarind pulp was prepared and used as a greener catalyst for the rapid and efficient synthesis of 2,4,5-trisubstituted imidazole by a three-component, one-pot condensation of 1, 2-diketones and aryl aldehydes in excellent yield under solvent-free conditions using microwave irradiation. 1*H*-imidazoles were known for their medicinal and pharmaceutical applications. These derivatives were found to be very active against pathogens. The crude products were isolated by filtration and purified by recrystallisation in ethanol. Structural confirmation was performed using melting point determination, FTIR, and $^{1}$H NMR spectroscopy. FTIR spectra revealed characteristic bands for aromatic, hydroxyl, and carbonyl functionalities, while $^{1}$H NMR spectra confirmed the presence of aromatic protons consistent with the imidazole framework. The method demonstrates a sustainable approach to heterocyclic synthesis by utilising renewable natural resources and energy-efficient techniques, aligning with the principles of green chemistry.

**KEYWORDS:** Microwave irradiation; Tamarind; solvent-free conditions; Three-component reaction.

## 1. INTRODUCTION

Imidazole is a five -membered heterocyclic compound containing two nitrogen atoms at non-adjacent positions. It has the molecular formula $C_3H_4N_2$ & is an important structure in many biological and chemical systems.

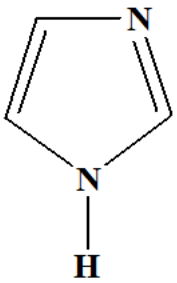



**Fig. 1. Structure of 1H- imidazole.**

Green synthesis of benzimidazole is a sustainable process for producing derivatives of benzimidazole. This artificial method reduces the process's environmental impact by minimizing the use of dangerous chemicals, hazardous solvents, and excessive energy inputs. In order to produce high-yield and high-purity benzimidazole derivatives, the process uses natural or renewable resources as catalysts or reagents, such as plant extracts or biological waste. Benzil, ammonium acetate, and a variety of aromatic aldehydes have been used to create a straightforward and effective generic process for the three-component reaction methodology of 2,4,5-trisubstituted imidazole. Imidazole is typically synthesized using environmentally hazardous chemicals, toxic catalysts, and harsh reaction conditions. Consequently, there is a growing interest in sustainable chemistry for effective and environmentally friendly synthesis.

In this context, *Tamarindus indica* pulp is used as natural organic acid is a tropical tree known for its culinary, medicinal, and industrial application. As Tamarind contain tartaric acid which contribute to its sour taste. It is an alternative for various chemicals as well as eco-friendly, cost-effective, widely available, non-toxic. These acids not only provide an acidic environment to facilitate the reaction but may also act as hydrogen bond donors, stabilizing transitions states and intermediates.

In order to achieve high efficiency, little use of hazardous reagents, and less influence on the environment, this work investigates the one-pot synthesis of imidazole derivatives using natural organic acid as catalysts. The strategy emphasises atom economy, sustainable catalysts, and decreased waste output, all of which are in line with the tenets of green chemistry.

**Review of Literature**

Imidazole is a nitrogen-containing heterocyclic ring with five members that is widely used in material science, agrochemicals, and medicines. Because of their important biological effects, imidazole and its derivatives are essential in many domains, including pharmaceutical chemistry. Greener and more effective methods have been developed because traditional synthesis procedures for imidazole derivatives sometimes entail hazardous conditions and harmful chemicals.

Recent advancements have focused on utilising natural organic acids and environmentally benign catalysts to synthesise imidazole derivatives.

For example, a solvent-free, catalyst-free one-pot technique for synthesising 1,2,4,5-tetrasubstituted imidazole's was presented in a work by Pranab Jyoti Das et al. [1]. This method effectively produces the necessary chemicals by reacting Benzil, aromatic aldehydes, ethanolamine, and ammonium acetate at 120°C.

Boric acid was used as a green catalyst in aqueous media under ultrasonic irradiation by Gurumeet C. Wadhawa et al. [2]. Excellent yields of 2,4,5-triaryl-1H-imidazoles can be produced at room temperature by condensing Benzil or benzoin, aldehydes, and ammonium acetate in a single pot. Second, boric acid ($BO_3H_3$) is a cheap, mild, and effective catalyst. This method's outstanding benefits include a green catalyst, gentle reaction conditions, straightforward processes, significantly faster reactions, and an exceptional product yield.

To create highly effective and metal-free tri- and tetra-substituted imidazole's for the synthesis of 2,4,5-triaryl-1H-imidazoles in excellent yields from the one-pot three-component condensation of Benzil/benzoin, an aldehyde, and ammonium acetate in aqueous media under ultrasound at room temperature, Abu Zafar Al Munsur et al. [3] used 3-picolinic acid as a catalyst. Additionally, the four-component synthesis of 1,2,4,5-tetra-substituted imidazole has been made possible by this catalyst. Easy purification, cost-effectiveness, high yielding, and most importantly, an environmentally safe approach are the main benefits of this technology.

Schiff bases were employed in the synthesis of imidazole derivatives by Divya Singh, Rajeev Kharb, and Satish Kumar Sharma [4]. Spectroscopic techniques were used to synthesise and

characterise a number of substances. Potential active compounds were found using in-silico docking analysis. TNF-α and IL-1β β were measured as inflammatory cytokines after the anti-inflammatory qualities of these compounds were examined using a carrageenan-induced paw oedema model. According to the results, certain Schiff's base imidazole derivatives have the potential to be effective anti-inflammatory treatments.

These methodologies underscore the shift towards more sustainable practices in organic synthesis, emphasising the use of natural acids, benign catalysts, and energy-efficient techniques to achieve efficient and green synthesis of imidazole derivatives.

Imidazole and its derivatives are a prominent class of heterocyclic compounds known for their diverse and potent biological activities. The imidazole ring is a key structural motif in many naturally occurring molecules, such as histidine and histamine, and is present in several clinically important drugs. The biological activity of imidazole's is attributed to their ability to bind to enzymes, metal ions, or biological receptors, thereby modulating various physiological processes. Because of this wide spectrum of activity, imidazole derivatives continue to be a rich area of research in medicinal chemistry and drug development. recent progress in the development of imidazole-containing derivatives as anti-tubercular agents and to summarise their structure-activity relationships. A deeper understanding of these relationships may facilitate the rational design of more effective imidazole-based anti-tubercular agents [5].

Derivatives of imidazole are widely used to treat fungal infections. Important enzymes in fungal cell membranes are inhibited by medications such as clotrimazole and miconazole. More recent substances demonstrated strong efficacy against fungus such as Aspergillus niger and Candida albicans, particularly those containing nitro groups or thiazolidinone rings. These results encourage more investigation into modified imidazole antifungals. [6] Additionally, imidazole compounds are effective against both Gram-positive and Gram-negative bacteria. Combining them with recognised antibacterial structures like triazoles and pyrazoles or with metal ions like silver, copper, or cobalt increases their antibacterial potency. These combinations help overcome antibiotic resistance and broaden the activity spectrum. [7] Martino et al. have conducted research in the laboratory & identified various imidazole derivatives exhibiting broad-spectrum chemotherapeutic properties. inquiry and galvanised by the work of de, we embarked on the current investigation to synthesise and evaluate novel imidazole's and nitroimidazoles capable of suppressing HIV replication while also inhibiting opportunistic microorganisms. [8] Numerous imidazoline-containing compounds have been shown to provoke insulin secretion from isolated pancreatic islets and, crucially, enhance

glucose tolerance in both rats and mice. [9] Imidazole and benzimidazole derivatives are associated with a broad spectrum of biological activities, including anti-inflammatory effects; various N-substituted imidazole is and substituted imidazolones exhibit notable anti-inflammatory properties. [10, 11]

## II. METHOD & MATERIALS

### (a) Material

All the chemicals and reagents utilised were of analytical and laboratory quality, and they weren't further purified. Ammonium acetate, Benzil, aromatic aldehyde, tamarind pulp, beaker, glass rod, microwave, watch glass, ethanol, n-hexane, and ethyl acetate are among the materials utilised.

### (b) Method

#### (i) Preparation of Tamarind Pulp

Ripe tamarind was chosen in order to make tamarind pulp. The pulp was then removed from the seeds, fibres, and shell. 50g of tamarind pulp were cooked after being soaked in water at a 1:2 ratio. A smooth tamarind pulp was produced by filtering the cooling mixture through muslin fabric to get rid of any solids.

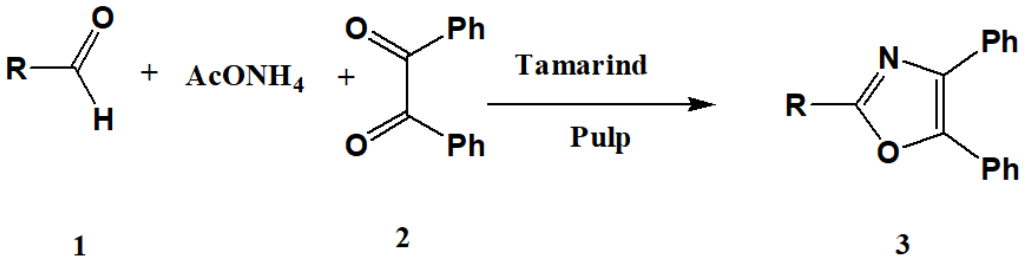
**(ii) General Procedure:** Benzil, ammonium acetate, tamarind pulp, and aromatic aldehyde were combined in a beaker and thoroughly mixed. Following that, the combination was exposed to microwave radiation. TLC tracked the progression of the reaction. The product was obtained by cooling the combination, neutralising it with 5% sodium bicarbonate, diluting it with water, and then pouring it over crushed ice. After filtering and washing, the solid was recrystallised from ethanol. Melting point, IR, $^{1}H$ NMR, and $^{13}C$ NMR spectroscopy were used to describe the pure chemical.

#### (iii) Experimental Procedure

A 50 ml Borosil beaker was filled with a mixture of aromatic aldehyde, Benzil (2.10 g, 210.23 g/mol), ammonium acetate (0.8 g, 77.084 g/mol), and 2 g of tamarind pulp. To obtain the desired result, the reaction mixture was agitated and put in a microwave. Thin Layer Chromatography (TLC) was utilised to monitor the product, and a suitable solvent mixture served as the eluent. The reaction mixture was allowed to cool to room temperature once the reaction was finished. After that, it was diluted with water, neutralised with a 5% $NaHCO_3$ solution, and then poured over crushed ice. To create a purified chemical, the resulting crude solid product was filtered, dried, and recrystallised using ethanol.

Further analysis was carried out to determining the melting point, and spectroscopic data were recorded using $^{1}$H- NMR,$^{13}$C-NMR, IR Spectroscopy.

**Schematic Study**

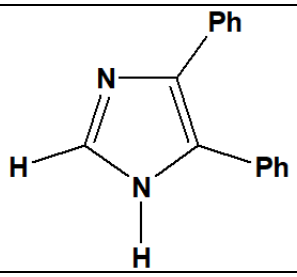
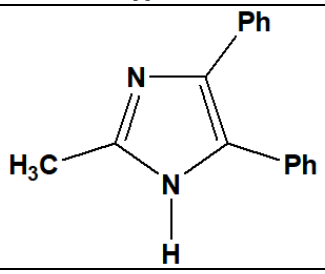
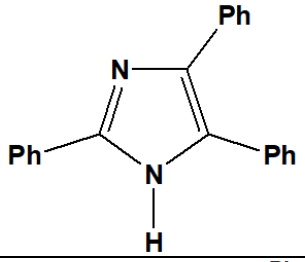
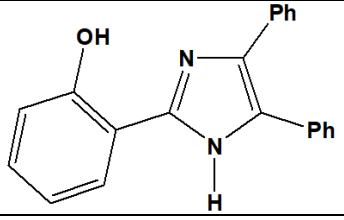


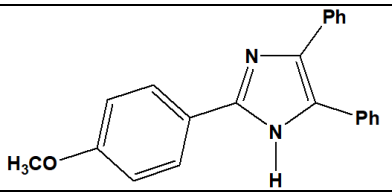
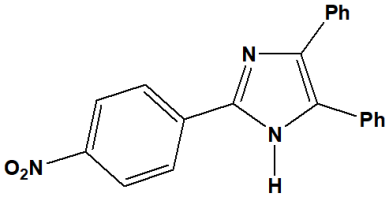
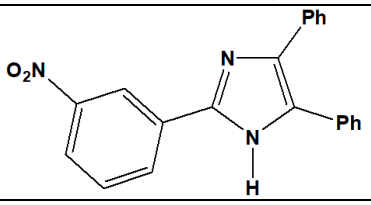
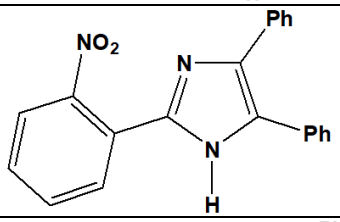
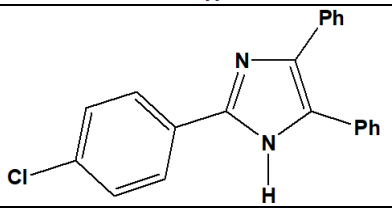

**1** **2** **3**

**Where R = Aryl or Alkyl**

## III. RESULTS & DISCUSSION

**Table 1. Physical data of newer imidazole derivatives using tamarind pulp.**

| Entry | Structure | Time in Minutes | Percentage yield (%) | Physical constant(°C) |
|---|---|---|---|---|
| 3a |  | 2 | 75 | 80 |
| 3b |  | 0.5 | 70 | 90°C |
| 3c |  | 1 | 85 | 70°C |
| 3d |  | 0.5 | 60 | 83°C |

| | | | | |
|---|---|---|---|---|
| **3e** |  | **1** | **85** | **65°C** |
| **3f** |  | **0.9** | **72** | **80°C** |
| **3g** |  | **0.4** | **55** | **90°C** |
| **3h** |  | **0.3** | **65** | **100°C** |
| **3i** |  | **1** | **78** | **98°C** |

**Characterization & Interpretation**

**Spectral Data: FTIR ($cm^{-1}$ )**

The FTIR spectrum of Sample 3b (1215) indicates the presence of several key functional groups. A broad peak at 3547 $cm^{-1}$ suggests an O–H stretch, indicating either an alcohol or phenol. Peaks at 3062 $cm^{-1}$ and around 1589–1447 $cm^{-1}$ are characteristic of aromatic C–H stretching and C=C ring vibrations, confirming the presence of an aromatic ring. Aliphatic C–H stretching is observed at 2931 and 2879 $cm^{-1}$. A strong absorption at 1699 $cm^{-1}$ corresponds to a C=O stretch, pointing to a carbonyl group such as in a ketone, ester, or carboxylic acid. The peaks between 1200 and 1000 $cm^{-1}$ (notably at 1169 and 1069 $cm^{-1}$) indicate C–O stretching, supporting the presence of alcohols, esters, or ethers. Additional bands near 715 and 872 $cm^{-1}$ correspond to out-of-plane aromatic C–H bending. Altogether, the spectrum suggests that the compound likely contains an aromatic ring, a carbonyl group, and either a hydroxyl or ether/ester functionality.

**$^1$H-NMR**

The provided $^1$H NMR spectrum displays distinct proton signals across a chemical shift range of 0 to 13 ppm. The most prominent region in the spectrum lies between δ 6.5 and 8.5 ppm, where multiple sharp peaks are observed. This region is characteristic of aromatic protons and suggests the presence of a substituted aromatic ring system, such as a phenyl group or a heterocyclic structure like imidazole. The multiplet pattern of these peaks indicates spin-spin coupling between adjacent protons on the ring. Given the nature of your project—imidazole derivatives—this aromatic region likely corresponds to the C2–H, C4–H, and C5–H protons of the imidazole ring and any additional aromatic substituents.

In the downfield region, between δ 10 and 12 ppm, there may be a broad singlet (though faint in this spectrum), which typically corresponds to an exchangeable proton like that of a carboxylic acid (–COOH), amide (–NH), or hydrogen-bonded –OH. Such protons are usually deshielded due to hydrogen bonding and the electron-withdrawing nature of nearby groups. Further up field, in the range of δ 2 to 5 ppm, small peaks are visible which may correspond to aliphatic protons adjacent to electronegative atoms (like –$CH_2$ next to nitrogen or oxygen) or possibly methylene/methyl groups in the side chain. Additionally, any solvent peaks should be taken into account: for example, DMSO-$d_6$ shows a peak at δ 2.5 ppm, and $CDCl_3$ at δ 7.26 ppm, which might overlap with sample signals if not subtracted. Overall, the spectral data supports the presence of an aromatic or heteroaromatic compound—consistent with an imidazole derivative—possibly bearing functional groups like –OH, –NH, or –COOH.

The present work demonstrates an efficient and green approach for synthesising imidazole derivatives using tamarind pulp—a natural organic acid—as a catalyst under microwave-assisted conditions. The reaction involved a one-pot condensation of benzil, an aromatic or aliphatic aldehyde, and ammonium acetate, with tamarind pulp providing a mildly acidic, eco-friendly reaction medium.

Microwave irradiation significantly accelerated the reaction, with completion times ranging from 30 seconds to 2 minutes depending on the substituents on the aldehyde. The reaction progress was monitored by TLC, and products were isolated in good purity after simple filtration and recrystallisation from ethanol. Melting points were recorded as physical constants to confirm purity and consistency.

The synthesised compounds displayed varied melting points (physical constants) ranging from 65°C to 90°C, influenced by the electronic nature of substituents on the aromatic ring. Electron-donating groups (e.g., methoxy, methyl) generally led to lower melting points, while

nitro-substituted derivatives showed higher melting points (e.g., 90°C), indicating stronger intermolecular interactions due to increased polarity or rigidity.

Overall, this study demonstrates that tamarind pulp can be used as an effective natural acid catalyst for imidazole synthesis, offering a green, cost-effective, and time-saving alternative to conventional methods. The rapid reaction times and simple purification steps, combined with reliable spectral characterisation, confirm the success of this environmentally benign protocol.

## IV. CONCLUSION

In this study, tamarind pulp, a naturally occurring organic acid, was used as a bio-catalyst under microwave irradiation to create imidazole derivatives in an environmentally friendly and effective manner. Without the use of hazardous solvents or artificial acids, the reaction was finished in a short amount of time (between 30 and 2 minutes), making the procedure energy-efficient and environmentally benign. The process had a number of benefits, such as low cost, quick reaction times, convenience of use, and high final product purity. The effective production of imidazole structures was confirmed by determining the melting point of the synthesised compounds and characterising them with FTIR and $^{1}$H NMR spectroscopy. This approach highlights the potential of using renewable natural materials for sustainable organic synthesis and aligns well with the principles of green chemistry.

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