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Tamarind Juice Assisted Benign Synthesis Of 2,3-Dihydro-1h-Perimidine Derivatives

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ABSTRACT: A green and efficient way to make 2,3-dihydro-1H-perimidine derivatives is through a one pot reaction of 1,8-diaminonaphthalene with different aromatic aldehydes using tamarind (Tamarindus indica L.) juice. The reactions were carried out in mild conditions, giving us the desired products in good or excellent yields with short reaction times. Tamarind juice was inexpensive, and easily available, and it was a metal-free and eco-friendly catalyst. The protocol is easy to follow, easy to separate products, low amount of catalyst, and does not include hazardous chemicals and chromatographic purification. This green way shows that tamarind juice is a good green catalyst for biologically important perimidine derivatives.

KEYWORDS: Tamarind Juice, Biocatalyst, Perimidine, Heterocyclic chemistry, One-pot Synthesis.

INTRODUCTION

The development of sustainable and environmentally friendly synthetic techniques has been encouraged by growing worries about environmental contamination and the loss of natural resources. Green chemistry promotes safer, more cost-effective, and efficient chemical processes while reducing the use and production of hazardous materials. Using renewable, biodegradable, and ecologically safe substitutes for hazardous solvents and corrosive catalysts is a crucial strategy. In this sense, naturally occurring biocatalysts and aqueous reaction media have become important resources for sustainable organic synthesis. Fruit extracts, owing to their low cost, easy availability, environmental safety, and catalytic efficacy, have received much appreciation as green catalysts in recent years. Fruit juices like those of lemon, pineapple, coconut, and tamarind have been used effectively as catalysts in reactions involving Biginelli condensation, Knoevenagel–Michael addition, and the formation of heterocycles. Tamarind (Tamarindus indica L.), belonging to the family Fabaceae, can be considered an excellent natural catalytic system for organic reactions. The fruit of tamarind possesses substantial amounts of organic acids, especially tartaric acid with small amounts of citric, malic, and ascorbic acids, giving rise to acidity of the aqueous extract (pH ≈ 3) of tamarind. Other components of tamarind fruit include carbohydrates, flavonoids, polyphenols, vitamins, and minerals [1-12].

The nitrogen heterocyclic compounds constitute one of the most significant groups of substances in medicinal and pharmaceutical chemistry. One such group of substances is the perimidine derivatives, which have received much interest owing to their numerous biological and physicochemical properties. The perimidines are usually prepared by the condensation reaction of 1,8-diaminonaphthalene with aldehyde and ketones. The perimidines possess several biological activities like antimicrobial, antifungal, antiviral, anticancer, anti-inflammatory, and antioxidant activities. Moreover, due to their unique properties, perimidines can be used as fluorophores, anticorrosion agents, chemosensors, dyes, and other materials. A number of different strategies for preparing perimidine derivatives have been developed. However, the majority of these involve the use of harsh reagents such as strong acids, metals, elevated temperature, and toxic organic solvents, thus increasing production costs as well as causing additional harm to the environment. Consequently, an environmentally friendly approach towards the synthesis of perimidine derivatives would be very beneficial [13-24].

The wide range of perimidine derivatives highlights the importance of the perimidine nucleus in synthetic, biological, medicinal, and other fields of chemistry. Representative perimidine derivatives and their structures are summarized below.

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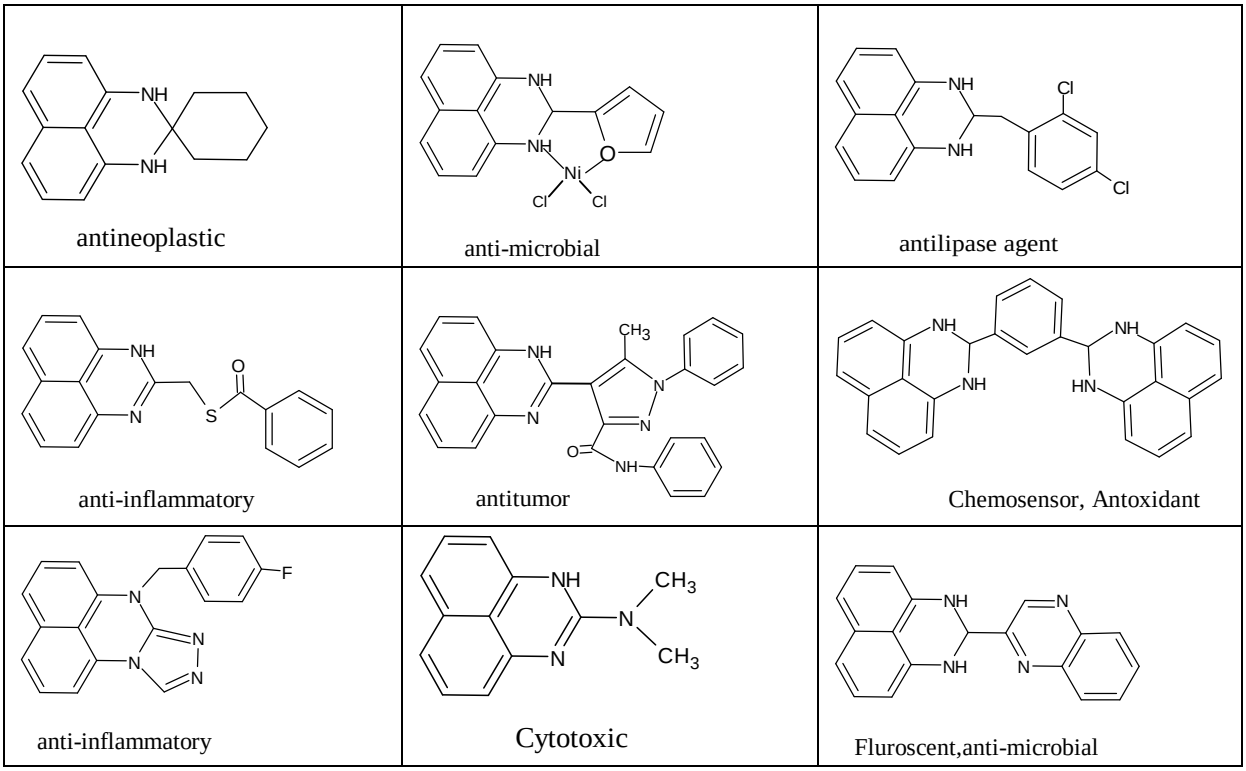

Here, we report the environmentally friendly synthesis of four different derivatives of perimidine using the natural biocatalyst from the extract of tamarind fruits by condensation of 1,8-diaminonaphthalene with aromatic aldehydes.

EXPERIMENTAL

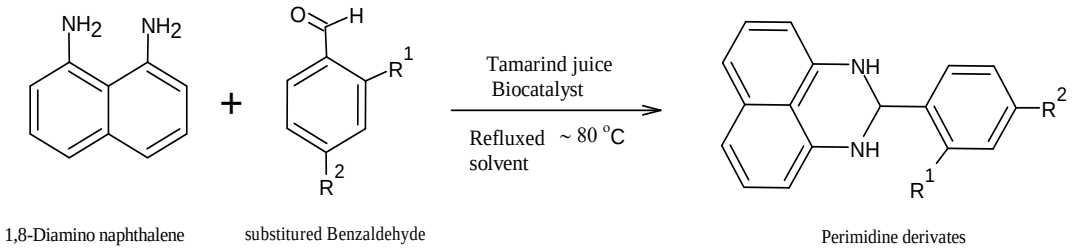

Figure1. Reaction scheme of synthesis of Perimidine derivatives

MATERIALS AND METHODS

Sigma-Aldrich, E. Merck (India), SDFC Ltd., and Loba Chemie provided all the necessary chemicals, which were used exactly as specified. The melting point ranges of newly synthesized compounds were determined by open glass capillary tubes. Thin layer chromatography (TLC) on aluminum plates coated with a layer of silica gel, provided by Merck, was used to assess the purity of prepared compounds and the progress of all reactions. An iodine chamber was used to visualize the spots. Using KBr pellets, the Shimadzu FTIR model 4800S spectrophotometer captured the infrared spectra in the 4000-400 cm^{-1} region. ^1H NMR spectrum was recorded in DMSO-d_6 on Bruker AV-300 (300 MHz) using TMS as an internal standard.

Preparation of Tamarind Fruit Extract (Biocatalyst)

Fresh tamarind (*Tamarindus indica*) fruits were purchased from a local market. The outer shell and seeds were removed manually, and 10 g of tamarind pulp was collected. The pulp was soaked in 50 mL of distilled water for 5 h to facilitate the extraction of water-soluble constituents. Subsequently, the mixture was heated with stirring for 2 h to obtain a homogeneous extract. After

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cooling to room temperature, the mixture was filtered twice to remove insoluble matter and then centrifuged using a microcentrifuge. The clear aqueous extract obtained was found to have a pH ≈ 3.0 due to the presence of naturally occurring organic acids, mainly tartaric acid along with citric and malic acids. The freshly prepared tamarind extract was stored at 3-5 °C and utilized as a green biocatalyst for the synthesis of perimidine derivatives [25, 26].

General Procedure for the Synthesis of 2-Phenyl-2,3-dihydro-1H-perimidine and its derivatives

A mixture of substituted benzaldehydes/benzaldehydes (0.01 mol), and 1,8-diamino naphthalene (0.01 mol) in a minimum amount of ethanol was stirred in presence of 10 mL tamarind juice (pH≈3.0) for few minutes at room temperature and then refluxed for appropriate time as mentioned in Table 2. The progress of the reaction was monitored by thin layer chromatography (TLC) technique. After completion of the reaction the reaction mixture was concentrated under vacuum. The solid formed on cooling was filtered off, washed several times with a minimum amount of ice-cold water and the crude product thus obtained. Finally, the crude product was recrystallized with ethanol to give a pure product and dried in a vacuum desiccator over anhydrous $CaCl_2$. The melting points of all synthesized compounds were well reported and identified.

RESULTS AND DISCUSSION

The model reaction of benzaldehyde with 1,8-diaminonaphthalene was chosen to explore a green and environmentally benign protocol for the synthesis of perimidine derivatives. The reaction was carried out under reflux condition for 1h with tamarind juice as natural biocatalyst (Scheme 1). Benzaldehyde was chosen as the first probe aldehyde to optimize the catalyst loading. After an hour of refluxing, the reaction conducted in ethanol alone without the addition of tamarind juice yielded very little product, suggesting that a catalyst is needed to produce a desired product (Entry 1, Table 1). The reaction was then performed with different volumes of tamarind juice (2-10 mL). The catalyst loading was found to have a significant effect on the product yield. Using 2 mL of tamarind juice, the desired product 2-phenyl-2,3-dihydro-1H-perimidine was obtained in 22% yield (Entry 2, Table 1). Increasing the amount of the catalyst to 4 mL improved the yield to 40% (Entry 3, Table 1). Tamarind juice (6 mL) was used and the product was obtained in 64% yield (Entry 4, Table 1).Further increase in the catalyst loading to 8 mL gave a substantial increase in the yield of the product to 80% (Entry 5, Table 1). The yield was 82 % (Entry 6, Table 1) with 10 mL of tamarind juice. When the quantity of catalyst is increased from 8 mL to 10 mL, the yield rises from 80% to 82%. This suggests that optimum conditions are attained during the course of the reaction. Melting point, FT-IR, and NMR analyses of the final product revealed that the results matched those published in the literature [27, 28]. This validates that 2-phenyl-2,3-dihydro-1H-perimidine was synthesized and the tamarind juice used as a biocatalyst was effective.

Table 1. Optimization of Tamarind Juice Amount for the Synthesis of Perimidine Derivatives

Entry	Catalyst Loading	Volume (mL)	Time (min.)	Yield (%)
1	Tamarind Juice	0	60	No reaction
2	Tamarind Juice	2	60	22
3	Tamarind Juice	4	60	40
4	Tamarind Juice	6	60	64
5	Tamarind Juice	8	60	80
6	Tamarind Juice	10	60	82

Figure 2 illustrates a possible mechanism for the synthesis of perimidine compounds accelerated by tamarind juice. First, protonation activates the carbonyl group of the aromatic aldehyde (1), producing intermediate (2) and making the carbonyl carbon more electrophilic. The activated carbonyl carbon is subsequently attacked by one amino group of 1,8-diaminonaphthalene (3) to create the carbinolamine intermediate (4), which deprotonates to produce the intermediate (5). The second amino group's intramolecular nucleophilic attack causes cyclization and the formation of an intermediate (7). Finally, the required perimidine derivative is obtained through deprotonation (8). Tamarind juice promotes the reaction effectively under mild conditions with good yield and selectivity by facilitating the substrate activation and proton transfer phases.

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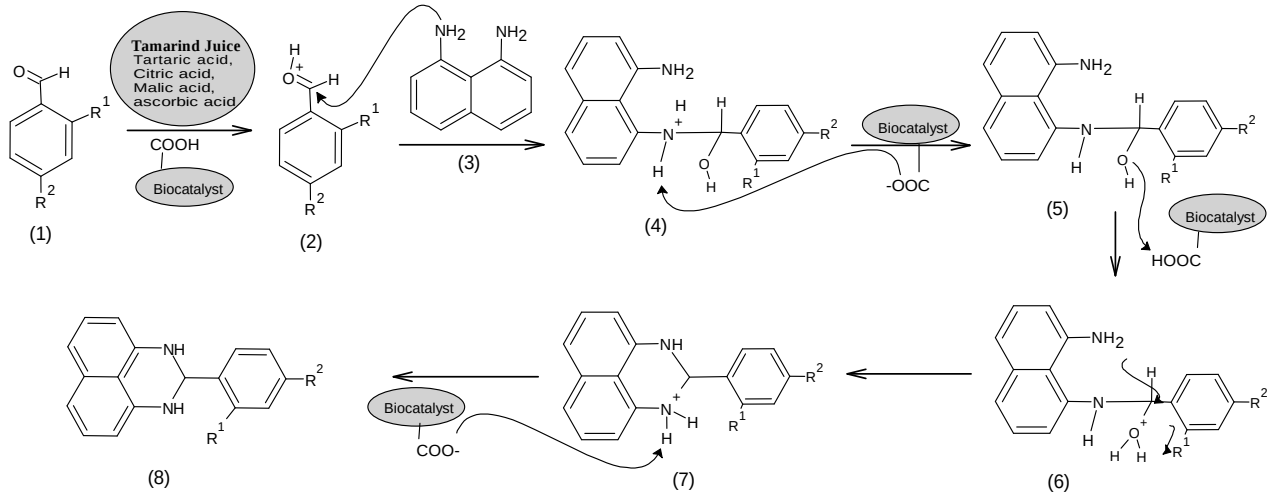
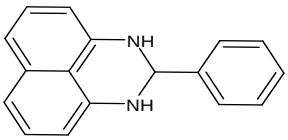
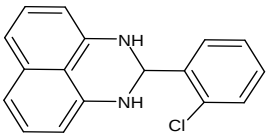
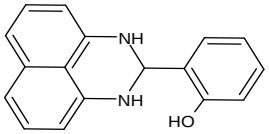


Figure 2. Possible mechanism for the Synthesis of Perimidine derivatives using Tamarind juice

Based on the optimization experiment, it was found that an amount of 10 mL of tamarind extract could be used to effectively catalyze the reaction in a very short period of time. An increase in catalyst loading did not contribute to a notable yield improvement. Accordingly, an amount of 10 mL was chosen as an optimum amount of catalyst loading for further reactions. With optimized conditions, different types of aromatic aldehydes having electron-donating or electron-withdrawing groups easily reacted with 1,8-diaminonaphthalene to produce the corresponding perimidine derivatives in good to excellent yields.

Table 2. Optimization study of Substrate for the Synthesis of Perimidine Derivatives

Sr. No.	Aldehyde	Product/Name	Time (min.)	Yield (%)	Melting point (^{0}C)	
					Observed	Lit. value
1	R^1=H, R^2 = H	 2-Phenyl-2,3-dihydro-1H-perimidine	45	82	103-105	101-103 [27]
2	R^1 = Cl, R^2 = H	 2-(2-chlorophenyl)-2,3-dihydro-1H-perimidine	40	83	118-120	117-118 [28]
3	R^1 = OH, R^2 = H	 2-(2,3-dihydro-1H-perimidine) phenol	55	78	192-194	189-191 [27]

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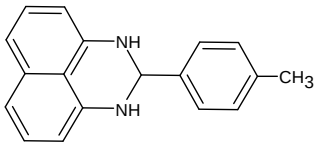
4	R^1 = H, R^2 = CH_3	 2-(4-Methylphenyl)-2,3-dihydro-1H-perimidine	60	75	164-166	166-167 [28]

2-Phenyl-2,3-dihydro-1H-perimidine:

IR: 3380, 3356 ν(N-H), 3040, 2805 ν(C-H), 1600 ν(C=C), 1420, 1260, 815, 759, 708.

^{1}H-NMR (DMSO-d_6): 5.35 (s, 1H, CH), 7.62–6.48 (m, 13H, 11C_{Ar}-H, 2 NH).

CONCLUSION

This paper presents the synthesis of perimidine-based derivatives using tamarind juice as a natural biocatalyst. The efficacy of tamarind juice as a biocatalyst to facilitate reactions allows for efficient formation of perimidine derivatives. Tamarind juice has many advantages such as being easily accessible and being environmentally friendly; which results in shorter reactions and ease of product separation makes this approach a significant example of a viable green method for the synthesis of perimidine derivatives.

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