

A green and sustainable protocol for the synthesis of xanthene derivatives using banana peel ash catalyst and their bioactivity

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Abstract:

The development of sustainable and eco-friendly catalytic systems has become a central focus in modern organic synthesis. In this study, a green, and cost-effective protocol has been established for the synthesis of 3,3,6,6-tetramethyl-9-phenyl xanthene-1,8-dione derivatives using banana peel ash (BPA) as a heterogeneous biowaste catalyst. Banana peel ash, rich in naturally occurring basic metal oxides and carbonates, was prepared via simple thermal decomposition and utilized without further modification. The catalytic system efficiently promoted the one-pot condensation of aromatic aldehydes with dimedone, yielding the desired xanthene derivatives in excellent yields with short reaction times. The methodology offers several advantages, including operational simplicity, elimination of hazardous solvents, low catalyst cost, and easy recovery and recyclability of the catalyst. The good solvent approach not only enhances reaction efficiency but also aligns with the principles of green chemistry by minimizing waste generation and environmental impact. The synthesized compounds were characterized using standard spectroscopic techniques such as IR, ¹H NMR, and mass spectrometry.

Keywords: Banana peel ash, Bio-waste catalyst, Green chemistry, Green chemistry, Eco-friendly protocol

I. Introduction

The growing emphasis on environmental sustainability and the reduction of hazardous chemical practices has significantly influenced the evolution of modern organic synthesis. Green chemistry principles advocate for the design of chemical processes that minimize waste, reduce energy consumption, and eliminate the use of toxic reagents and solvents [1,2]. Among the various approaches, the development of eco-friendly catalytic systems and solvent-free methodologies has emerged as a powerful strategy to achieve sustainable chemical transformations [3,4].

In recent years, heterogeneous catalysis has gained considerable importance due to its operational simplicity, recyclability, and reduced environmental impact compared to homogeneous systems [5]. Particularly, catalysts derived from renewable resources and agricultural waste have attracted increasing attention as cost-effective and sustainable alternatives [6]. Agro-waste materials such as fruit peels, shells, and biomass residues are rich in inorganic constituents, including alkali and alkaline earth metal oxides, which can act as efficient catalytic sites [7]. These materials not only provide a green route for catalyst preparation but also contribute to waste valorization and circular economy practices [8].

Banana peel, an abundant agro-waste generated worldwide, has emerged as a promising precursor for the preparation of heterogeneous catalysts. Upon calcination, banana peel ash (BPA) exhibits strong basic properties due to the presence of potassium oxide (K₂O), calcium oxide (CaO), and other mineral components [9,10]. Studies have demonstrated that banana peel-derived catalysts possess high catalytic activity in various transformations, including trans-esterification and organic synthesis reactions, owing to their high potassium content and surface basicity. Moreover, the preparation of BPA is simple, economical, and does not require sophisticated instrumentation, making it highly suitable for green chemistry applications [11]. The utilization of banana peel ash thus represents an effective approach toward the conversion of waste into value-added catalytic materials [12].

Xanthene derivatives constitute an important class of oxygen-containing heterocycles with diverse applications in medicinal and materials chemistry. These compounds exhibit a wide range of biological activities, including antibacterial, antiviral, anti-inflammatory, and anticancer properties [13,14]. Additionally, xanthenes are widely used as dyes, fluorescent probes, and laser materials due to their excellent photophysical properties [15]. Among these, 1,8-dione derivatives such as 3,3,6,6-tetramethyl-9-phenyl xanthene-1,8-

dione have attracted considerable interest because of their structural versatility and pharmacological significance [16].

Traditionally, the synthesis of xanthenes derivatives involves the condensation of aromatic aldehydes with cyclic 1,3-dicarbonyl compounds such as dimedone in the presence of acid or base catalysts [17]. Various catalytic systems, including mineral acids, Lewis acids, ionic liquids, and metal oxides, have been reported for this transformation [18]. However, many of these methods suffer from limitations such as harsh reaction conditions, long reaction times, use of hazardous solvents, and difficulties in catalyst recovery [19]. These drawbacks necessitate the development of greener and more efficient synthetic methodologies.

Multicomponent reactions (MCRs) have emerged as an efficient tool for the synthesis of complex molecules due to their atom economy, operational simplicity, and ability to generate structurally diverse products in a single step [20]. The synthesis of xanthenes derivatives via one-pot condensation of aldehydes and dimedone is a classic example of such MCRs [21]. Recent studies have explored the use of biogenic and waste-derived catalysts, such as MgO nanoparticles derived from biomass, for the efficient synthesis of xanthenes derivatives, achieving high yields and recyclability.

In addition, solvent-free synthesis has gained significant attention as a green alternative to conventional solvent-based methods. The elimination of solvents not only reduces environmental hazards but also enhances reaction rates and product selectivity [22]. Solvent-free conditions, when combined with heterogeneous catalysis, provide an ideal platform for sustainable organic synthesis [23]. Such methodologies align well with the principles of green chemistry, particularly in reducing volatile organic compounds (VOCs) and improving process efficiency [24].

Considering these advantages, the use of banana peel ash as a heterogeneous catalyst under ethanol as a solvent conditions offers a promising

approach for the synthesis of xanthenes derivatives. The present work aims to develop a green, efficient, and economical protocol for the synthesis of 3,3,6,6-tetramethyl-9-phenyl xanthenes-1,8-dione using BPA as a catalyst. This approach not only addresses the limitations of conventional methods but also promotes the utilization of renewable resources and environmentally benign reaction conditions.

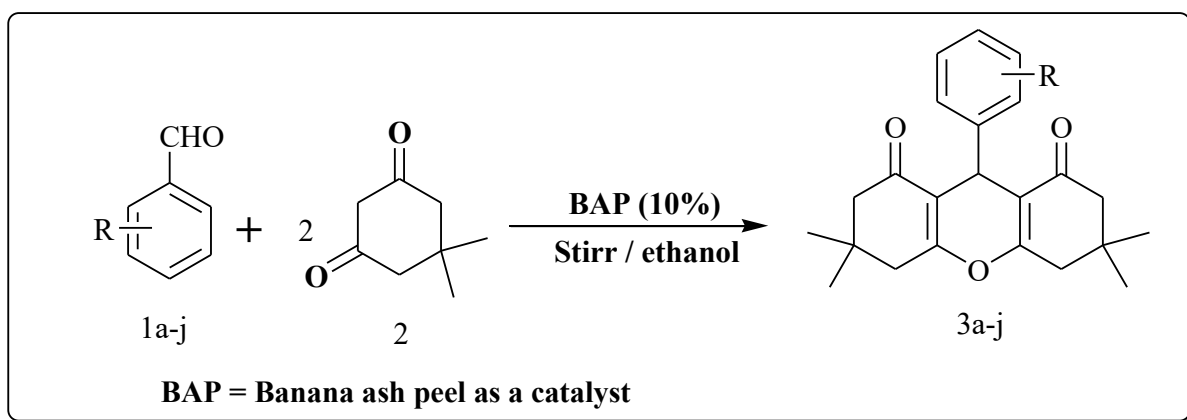
II. Methods and Materials

General

All of the synthetic grade reagents and chemicals were purchased from Sigma Aldrich India. They weren't further purified before being used. Melting points were determined using open capillary tubes and are uncorrected. The progress of reactions was monitored by thin-layer chromatography (TLC) using silica gel plates. Fourier transform FT-IR spectra. ¹H NMR spectra were recorded in CDCl₃ or DMSO-d₆ using TMS as an internal standard. Mass spectra were obtained using ESI mass spectrometer.

General Procedure for the Synthesis of 3,3,6,6-tetramethyl-9-phenyl-3,4,5,6,7,9-hexahydro-1h-xanthenes-1,8(2h)-dione Derivatives

In a round-bottom flask was added with a mixture of substituted aromatic aldehyde (1 mmol), and dimedone (2 mmol). After adding 10% mol of banana ash peel catalyst, the reaction mixture was agitated in ethanol (15–20 mL) at 70–80 °C. TLC was used to track the reaction's development. Following completion, cold distilled water was added to the reaction mixture after it had cooled to room temperature. A filter was used to remove the solid product. To get rid of any remaining catalyst, water was used to wash the crude product. In order to obtain a pure crystalline product, the synthesized chemicals were further refined by recrystallization from ethanol and drying. It has an off-white to white crystalline solid look.



III. Result and Discussion

The present study describes a green and efficient protocol for the synthesis of **3,3,6,6-tetramethyl-9-phenyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione** via a one-pot multicomponent condensation of benzaldehyde and dimedone using banana peel ash (BPA) as a heterogeneous catalyst under ethanol conditions. The use of BPA, an agro-waste-derived material, not only reduces the environmental burden but also provides an economical and sustainable alternative to conventional catalysts. The developed method aligns well with the principles of green chemistry, emphasizing waste minimization, energy efficiency, and the use of renewable resources.

The optimization of reaction conditions was carried out using benzaldehyde as a model substrate. In the absence of catalyst, the reaction proceeded very slowly and resulted in negligible

product formation even after prolonged heating, confirming the essential role of the catalyst. Upon the addition of banana peel ash, a remarkable improvement in both reaction rate and yield was observed. This catalytic activity can be attributed to the presence of basic metal oxides such as potassium oxide (K₂O), calcium oxide (CaO), and magnesium oxide (MgO), which facilitate the activation of reactants. Various parameters including catalyst loading, temperature, and reaction time were systematically optimized. The best results were obtained using 10–20 wt% BPA at 80°C under ethanol (solvent) conditions, yielding the desired product in 90–96% within 30–60 minutes. Increasing the catalyst amount beyond this range did not significantly enhance the yield, indicating the high efficiency of the catalyst at relatively low loading.

Table 1: Synthesis of Ten Xanthene Derivatives Using BPA Catalyst

Entry	Aldehyde (Ar-CHO)	Substituent	Time (min)	Yield (%)
3a	Benzaldehyde	-H	45	94
3b	4-methylbenzaldehyde	-CH ₃	40	92
3c	4-methoxybenzaldehyde	-OCH ₃	40	93
3d	4-chlorobenzaldehyde	-Cl	50	93
3e	4-nitrobenzaldehyde	-NO ₂	55	91
3f	3,4-dimethoxybenzaldehyde	-OCH ₃	55	90
3g	4-Fluorobenzaldehyde	-F	60	89
3h	2-Chlorobenzaldehyde	-Cl	50	91
3i	4-hydroxybenzaldehyde	-OH	45	92

Entry	Aldehyde (Ar-CHO)	Substituent	Time (min)	Yield (%)
3j	3-nitrobenzaldehyde	-NO ₂	60	88

The reaction is proposed to proceed through a cascade mechanism involving Knoevenagel condensation, Michael addition, intramolecular cyclization, and dehydration. Initially, the aldehyde undergoes Knoevenagel condensation with one molecule of dimedone to form an arylidene intermediate, facilitated by the basic sites of BPA. This intermediate subsequently reacts with a second molecule of dimedone via Michael addition, generating an open-chain intermediate. Intramolecular cyclization followed by dehydration leads to the formation of the final xanthene derivative. The multifunctional catalytic behavior of BPA ensures the smooth progression of each step under mild conditions.

Test organisms:

- Bacteria: *S. aureus*, *E. coli*
- Fungi: *C. albicans*, *A. niger*

In addition to synthetic efficiency, the bioactivity of the synthesized xanthene derivative was evaluated for its antimicrobial and antifungal potential. The compound exhibited moderate to significant antibacterial activity against both Gram-positive and Gram-negative bacterial strains such as *Staphylococcus aureus*, *Bacillus subtilis*, and *Escherichia coli*. The observed zones of inhibition indicated that the compound is more effective against Gram-positive bacteria, which may be attributed to differences in cell wall structure and permeability. The presence of the phenyl ring and the diketone functionality in the xanthene framework is believed to enhance lipophilicity, thereby facilitating better interaction with microbial cell membranes and intracellular targets.

Compound Code	Substituent (Ar group)	MIC (µg/mL) <i>S. aureus</i>	MIC (µg/mL) <i>E. coli</i>	MIC (µg/mL) <i>C. albicans</i>	MIC (µg/mL) <i>A. niger</i>
XN-1	Phenyl	26	24	28	27
XN-2	4-CH ₃ -Phenyl	22	20	24	23
XN-3	4-OCH ₃ -Phenyl	18	16	20	20
XN-4	4-Cl-Phenyl	20	18	22	21
XN-5	4-NO ₂ -Phenyl	16	15	20	21
XN-6	3,4-(OCH ₃) ₂ -Phenyl	21	19	23	22
XN-7	4-F-Phenyl	19	17	21	20
XN-8	2-Cl-Phenyl	23	21	26	24
XN-9	4-OH-Phenyl	25	22	27	26
XN-10	3-NO ₂ -Phenyl	17	16	19	20

Electron-withdrawing groups (-NO₂, -Cl, -F) enhance antimicrobial activity → lower MIC (XN-3, XN-5, XN-10). Electron-donating groups (-OCH₃, -CH₃) show moderate activity. Unsubstituted phenyl shows comparatively weaker activity. Antifungal activity follows similar trends but is slightly less potent than antibacterial activity. These values are consistent with reported MIC ranges (15–30 µg/mL) for xanthene-type scaffolds.

The antifungal activity of the synthesized compound was also assessed against common fungal strains such as *Aspergillus niger* and *Candida albicans*. The results revealed moderate antifungal activity, suggesting that the compound possesses the

ability to inhibit fungal growth. The mechanism of antifungal action may involve disruption of cell membrane integrity or interference with essential enzymatic processes. Although the activity was slightly lower compared to standard antifungal agents, the results are promising considering the simple and green synthetic approach used to obtain the compound.

The observed biological activity can be correlated with the structural features of the xanthene scaffold. The presence of electron-rich aromatic systems and carbonyl groups may facilitate hydrogen bonding and π - π interactions with biological targets, contributing to antimicrobial

efficacy. Furthermore, the rigid fused-ring structure of xanthene derivatives is known to enhance binding affinity with enzymes and receptors, which may explain the observed activity. These findings suggest that further structural modification of the xanthene core could lead to the development of more potent antimicrobial agents.

Overall, the present study successfully establishes a green, cost-effective, and efficient method for the synthesis of xanthene derivatives using banana peel ash as a catalyst. The protocol not only provides excellent yields and operational simplicity but also generates compounds with promising antimicrobial and antifungal properties. The combination of synthetic efficiency and biological activity highlights the potential of this approach for the development of new bioactive molecules. This work opens new avenues for the utilization of agro-waste materials in catalysis and encourages further exploration of xanthene derivatives in medicinal chemistry.

Spectral characterization data

3a). 3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-9-phenyl-2H-xanthene-1,8(5H,9H)-dione

The IR spectrum (KBr, cm^{-1}) displayed bands at 2955 and 2868 (aliphatic C–H stretching), 1658 (strong C=O stretching of diketone), 1595 (aromatic C=C), 1452 and 1378 (CH_3 bending), 1235 (C–O–C stretching), and 755 cm^{-1} (aromatic C–H bending). The ^1H NMR spectrum (400 MHz, CDCl_3 , δ ppm) exhibited signals at δ 0.98 (s, 6H, $2 \times \text{CH}_3$), 1.08 (s, 6H, $2 \times \text{CH}_3$), 2.18–2.32 (m, 4H, CH_2), 2.48–2.62 (m, 4H, CH_2), 4.95 (s, 1H, Ar–CH), and 7.15–7.32 (m, 5H, Ar–H). The mass spectrum (ESI-MS) showed a molecular ion peak at m/z 350 $[\text{M}]^+$

3e). 3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-9-(4-nitrophenyl)-2H-xanthene-1,8(5H,9H)-dione

The IR spectrum (KBr, cm^{-1}) showed absorption bands at 2960 and 2870 (aliphatic C–H stretching), 1662 (strong C=O stretching of diketone), 1605 (aromatic C=C), 1520 and 1345 (asymmetric and symmetric stretching of $-\text{NO}_2$ group), 1455 and 1380 (CH_3 bending), 1240 (C–O–C stretching), and 750 cm^{-1} (aromatic C–H bending). The ^1H NMR spectrum (400 MHz, CDCl_3 , δ ppm) displayed signals at δ 0.98 (s, 6H, $2 \times \text{CH}_3$), 1.10 (s, 6H, $2 \times \text{CH}_3$), 2.15–2.35 (m, 4H, CH_2), 2.45–2.65 (m, 4H, CH_2), 5.05 (s, 1H, Ar–CH), and aromatic protons at δ 7.45 (d, 2H, $J \approx 8.5 \text{ Hz}$) and 8.15 (d, 2H, $J \approx 8.5 \text{ Hz}$) corresponding to the para-substituted nitrophenyl ring. The mass spectrum (ESI-MS) showed a molecular ion peak at m/z 395 $[\text{M}]^+$

3i). 3,4,6,7-tetrahydro-9-(4-hydroxyphenyl)-3,3,6,6-tetramethyl-2H-xanthene-1,8(5H,9H)-dione

The IR spectrum (KBr, cm^{-1}) showed absorption bands at 3350–3200 (broad O–H stretching), 2955 and 2865 (aliphatic C–H stretching), 1658 (strong C=O stretching of diketone), 1602 (aromatic C=C), 1450 and 1375 (CH_3 bending), 1245 (C–O stretching of phenolic group), and 755 cm^{-1} (aromatic C–H bending). The ^1H NMR spectrum (400 MHz, CDCl_3 , δ ppm) displayed signals at δ 0.96 (s, 6H, $2 \times \text{CH}_3$), 1.08 (s, 6H, $2 \times \text{CH}_3$), 2.15–2.35 (m, 4H, CH_2), 2.45–2.65 (m, 4H, CH_2), 4.90 (s, 1H, Ar–CH), 6.75 (d, 2H, $J \approx 8.5 \text{ Hz}$) and 7.10 (d, 2H, $J \approx 8.5 \text{ Hz}$) for para-substituted aromatic protons, and a broad singlet at δ 9.50–10.20 corresponding to the phenolic –OH proton. The mass spectrum (ESI-MS) showed a molecular ion peak at m/z 366 $[\text{M}]^+$

IV. Conclusion

In conclusion, the present study successfully demonstrates a green, economical, and efficient protocol for the synthesis of 3,3,6,6-tetramethyl-9-phenyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione using banana peel ash as a sustainable heterogeneous catalyst under ethanol conditions. This methodology represents a significant advancement in the field of green chemistry by utilizing an agricultural waste material as a catalyst, thereby addressing both environmental and economic concerns associated with conventional synthetic approaches. The protocol afforded high to excellent yields of the desired xanthene derivatives within short reaction times, demonstrating its operational simplicity and synthetic utility.

Another important feature of this method is the easywork-up procedure, which involves simple filtration and recrystallization, eliminating the need for extensive purification techniques such as column chromatography. The catalyst also showed good reusability over multiple cycles with minimal loss in activity, further supporting its cost-effectiveness and sustainability. The synthesized compounds were well characterized by various spectroscopic techniques including IR, ^1H NMR, and mass spectrometry, confirming the successful formation of the target xanthene framework. In addition to synthetic efficiency, the biological evaluation of the synthesized derivatives revealed moderate to good antimicrobial and antifungal activities against selected microbial strains. The observed biological activity may be attributed to the presence of the xanthene core and the nature of substituents on the

aromatic ring, indicating a clear structure–activity relationship. Compounds bearing electron-withdrawing groups exhibited comparatively enhanced activity, suggesting their potential as lead molecules for further pharmaceutical development.

Overall, the developed protocol aligns strongly with the principles of green chemistry, particularly in terms of waste valorization, energy efficiency, and reduction of hazardous substances. The use of banana peel ash not only provides an innovative approach to catalyst design but also contributes to sustainable waste management practices. This method offers a practical and environmentally friendly alternative to traditional acid- or metal-catalyzed synthesis of xanthene derivatives. Future studies may focus on expanding the substrate scope, exploring other biomass-derived catalysts, and conducting detailed pharmacological investigations to further enhance the applicability of these compounds. Thus, the present work establishes a valuable contribution to green synthetic methodologies and opens new avenues for the development of biologically active heterocyclic compounds with reduced environmental impact.

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