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PREPARATION OF XANTHENE-DIONE DERIVATIVES BY 2-AMINOPHENOL UNDER SOLVENT FREE CONDITIONS

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ABSTRACT
The synthesis of xanthene-dione derivatives has gained significant attention due to their broad spectrum of biological and pharmaceutical applications. This study presents an efficient, eco-friendly, and solvent-free approach for the preparation of xanthene-dione derivatives using 2-aminophenol as a catalyst. The reaction involves a one-pot, three-component condensation of β-naphthol, aldehydes, and dimedone under controlled thermal conditions. The method offers high yields, operational simplicity, and eliminates the use of toxic solvents, aligning with the principles of green chemistry. Spectroscopic techniques such as IR, NMR, and Mass spectrometry were used to characterize the synthesized compounds, confirming their structural integrity. This sustainable synthesis protocol provides an environmentally benign alternative for the production of xanthene derivatives with potential applications in medicinal chemistry, fluorescence dyes, and material sciences.

Keywords: Xanthene-dione derivatives, 2-Aminophenol, Green Chemistry, Solvent-Free Synthesis, One-Pot Condensation, Spectroscopic Characterization.

INTRODUCTION
Xanthene and benzoxanthene derivatives have garnered considerable attention in organic synthesis due to their wide-ranging biological and pharmaceutical properties. These compounds exhibit significant therapeutic activities, including antiviral, anti-inflammatory, antibacterial, antifungal, anticancer, and antioxidant properties. Additionally,

they find applications in the preparation of dyes, laser technologies, biomolecular imaging, and pH-sensitive fluorescent materials. The structural versatility and bioactivity of xanthenes have made them a focal point for researchers seeking efficient and sustainable synthetic methodologies.

Traditional methods for synthesizing xanthene derivatives often require harsh reaction conditions, toxic metal catalysts, excessive reagent use, and environmentally hazardous organic solvents. To overcome these limitations, green chemistry principles have been integrated into modern synthetic approaches. One such method is the solvent-free synthesis of xanthene-dione derivatives using 2-aminophenol as a catalyst. This approach eliminates the need for toxic solvents, reduces reaction time, and enhances product yield, making it an eco-friendly alternative.

This research focuses on the one-pot, three-component synthesis of xanthene-dione derivatives via the condensation of β-naphthol, aldehydes, and dimedone under solvent-free conditions. The reaction is facilitated by 2-aminophenol, which acts as an efficient organocatalyst. The synthesized compounds are characterized using spectroscopic techniques such as IR, NMR, and Mass spectrometry to confirm their structural integrity and purity.

By adopting a sustainable and high-yielding synthesis method, this study aims to contribute to the development of green chemistry practices in pharmaceutical and material sciences. The solvent-free approach not only minimizes environmental impact but also provides a cost-effective and scalable method for the production of bioactive xanthene derivatives.

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I. METHODS AND MATERIAL

2.1 Materials

All reagents, including β-naphthol, aldehydes, dimedone, and 2-aminophenol, were of analytical grade and procured from standard chemical suppliers. The reactions were carried out without the use of solvents, adhering to green chemistry principles. High-purity ethanol was used for recrystallization and purification of the final products.

2.2 General Procedure for xanthene-dione derivatives Synthesis

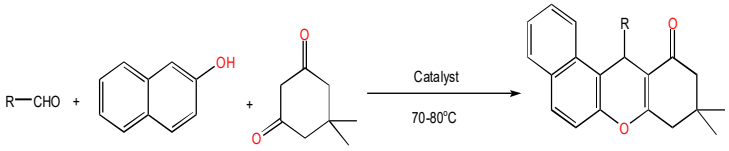
A mixture of β-naphthol (1.0 mmol), Aromatic aldehyde (1.0 mmol), and dimedone (1.0 mmol) was taken in a dry reaction vessel. 2-Aminophenol (5.0 mol%) was added as a catalyst, and the reaction was carried out under solvent-free conditions at 80°C with continuous stirring. The reaction progress was monitored using Thin Layer Chromatography (TLC).

After completion, 10 mL of cold distilled water was added to stop the reaction. The solid product was filtered, washed with distilled water, and recrystallized from hot ethanol to obtain pure xanthene-dione derivatives.

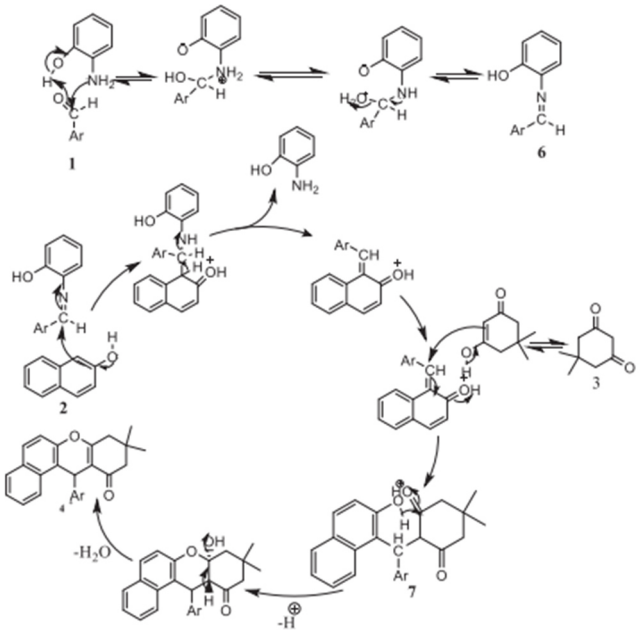
The final products were characterized using Infrared Spectroscopy (IR), Nuclear Magnetic Resonance (NMR), and Mass Spectrometry (MS) to confirm their structure and purity.

III. RESULTS AND DISCUSSION

3.1 Reaction



Mechanism :



The synthesis of xanthene-dione derivatives via a one-pot, three-component condensation reaction follows a sequential mechanism involving nucleophilic addition, condensation, and cyclization. The reaction proceeds as follows:

Step 1: Activation of Aldehyde

2-Aminophenol acts as an organocatalyst, activating the carbonyl group of the aldehyde through hydrogen bonding or weak coordination, increasing its electrophilicity.

Step 2: Nucleophilic Attack

β-Naphthol undergoes nucleophilic addition to the activated aldehyde, leading to the formation of an intermediate benzylidene compound.

Step 3: Enolate Formation

Dimedone, being a 1,3-diketone, exists in its enol form and reacts with the intermediate through a Michael addition or Knoevenagel condensation, forming a second intermediate.

Step 4: Intramolecular Cyclization

The newly formed intermediate undergoes cyclization via intramolecular nucleophilic attack, leading to the formation of the final xanthene-dione framework.

Step 5: Product Formation

The final product is stabilized through tautomerization and proton transfer, yielding highly functionalized xanthene-dione derivatives.

This solvent-free, metal-free approach provides an efficient, green, and high-yielding method for the synthesis of biologically active xanthene derivatives.

3.2 Product Characterization

The synthesized xanthene-dione derivatives were characterized using various spectroscopic techniques to confirm their structural integrity and purity.

1. Infrared Spectroscopy (IR)

C=O (Carbonyl Stretch): ~1683 cm^{-1}

C-O (Ether Stretch): ~1201 cm^{-1}

Aromatic C-H Stretch: ~3046 cm^{-1}

C-Br (Alkyl Halide, if applicable): ~816 cm^{-1}

2. Nuclear Magnetic Resonance (NMR) Spectroscopy

1H NMR (δ, ppm):

2.1–2.2 ppm: CH_3 (Methyl groups)

7.0–7.8 ppm: Aromatic protons (Benzene ring)

1.8 ppm: Additional CH_3 (if present)

13C NMR (δ, ppm):

168–170 ppm: Carbonyl (C=O)

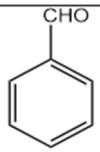
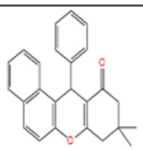
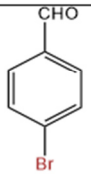
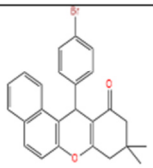
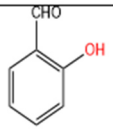
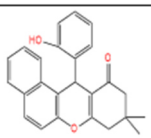
120–140 ppm: Aromatic carbons

3. Mass Spectrometry (MS)

Molecular Ion Peak (M^+) was observed, corresponding to the calculated molecular weight of the synthesized xanthene-dione derivative.
The fragmentation pattern confirmed the presence of the core xanthene-dione structure.

4. Melting Point Analysis
The melting point of the synthesized compounds was determined using a digital melting point apparatus to ensure purity and consistency with reported values.

The characterization results confirm the successful synthesis of xanthene-dione derivatives with high purity, supporting the efficacy of the solvent-free, green synthetic approach used in this study.

No	Aldehydes	Times (min)	Products	M.P	Yield (%)
1		25		120°C	60%
2		25		114°C	56%
3		25		112°C	50%

IR –

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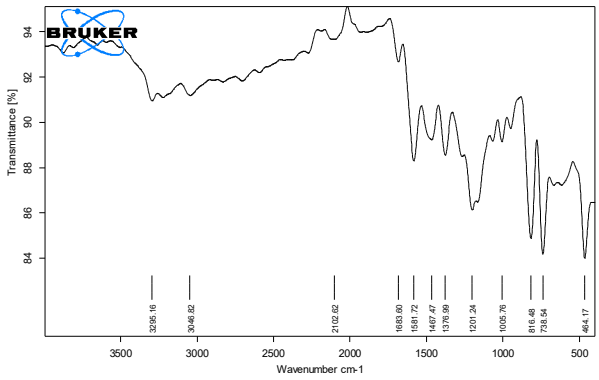
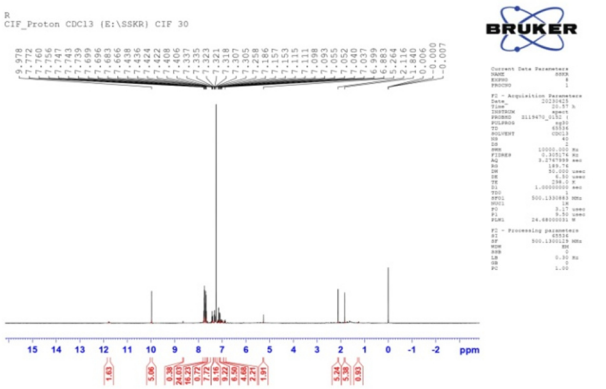
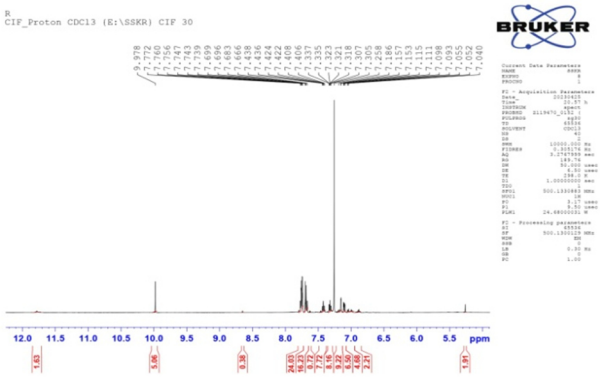


NMR :





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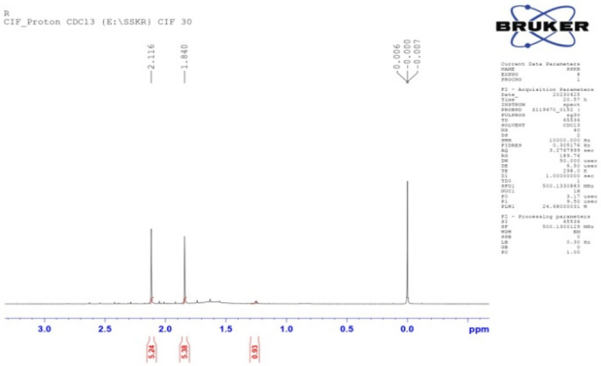

II. CONCLUSION

A solvent-free, eco-friendly method for synthesizing xanthene-dione derivatives using 2-aminophenol as a catalyst was successfully developed. The reaction proceeded efficiently under mild conditions, yielding high-purity products. Characterization by IR, NMR, and Mass Spectrometry confirmed the structural integrity of the synthesized compounds. This sustainable, metal-free approach offers a simple, high-yielding, and environmentally friendly alternative for xanthene derivative synthesis with potential pharmaceutical and industrial applications.

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