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Original Article

Reusable Solid Sulfonated Catalysts for the Effective Cyclisation of Heterocyclic Xanthene Derivatives

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Abstract

Sulphonated bagasse materials, and biological applications all make extensive use of xanthene derivatives, a significant class of heterocyclic chemicals. Reusable solid sulfonated catalysts are used in this study to synthesise heterocyclic xanthene derivatives in an effective and eco-friendly manner. Strong acidic sites found in sulfonated catalysts encourage condensation and cyclisation reactions between cyclic 1,3-dicarbonyl compounds and aromatic aldehydes. High catalytic activity, quick reaction times, good yields, and simple catalyst recovery are all features of the developed catalytic system. This method is a sustainable and environmentally friendly substitute for conventional homogeneous acid catalysts since the catalyst may be recycled multiple times without experiencing appreciable activity loss.

Keywords: *Sulphonated, bagasse, catalysts, recycled*

Introduction

In organic synthesis and medical chemistry, heterocyclic molecules are essential. Among these, xanthene derivatives have garnered a lot of interest because of their various industrial and biological uses, such as antiviral, antibacterial, anti-inflammatory, and anticancer properties [1-5]. They are also employed as laser dyes and fluorescent probes (6-7). Strong liquid acids like sulphuric acid or hydrochloric acid, which have disadvantages including corrosion, challenging separation, and environmental risks, are frequently used in traditional xanthenes synthesis processes [8-9]. Heterogeneous solid acid catalysts have evolved as effective and environmentally friendly substitutes to get around these restrictions (10). Strong Brønsted acidity and high catalytic activity are characteristics of solid sulfonated catalysts with $-SO_3H$ groups. Key organic transformations including condensation and cyclisation processes, which are necessary for the synthesis of xanthene, are facilitated by these catalysts (11). Heterogeneous catalysts also have benefits including ease of separation, recyclability, and less of an influence on the environment (12). According to recent research, sulfonated materials such magnetic sulfonated nanoparticles, cellulose-based catalysts, and sulfonated diatomite efficiently catalyse the one-pot synthesis of several xanthene derivatives with high yields in mild circumstances [13]. In the synthesis of xanthenes, solid sulfonated catalysts typically promote the acid-catalyzed condensation of aldehydes with phenolic compounds under mild conditions, leading to efficient formation of the xanthene ring system [14]. Their solid nature prevents corrosion issues and simplifies product purification [15]. To optimize the synthesis process using solid sulfonated catalysts, consider parameters such as catalyst loading, reaction temperature, solvent choice (or solvent-free conditions), and reaction time. Characterization of the catalyst's acid strength and surface area can also correlate with catalytic performance [16-17]. The selected text explains that in the synthesis of xanthenes, solid sulfonated catalysts facilitate the acid-catalyzed condensation of aldehydes with phenolic compounds under mild conditions, efficiently forming the xanthene ring system [18]. Their solid form offers advantages by preventing corrosion and simplifying purification (19).

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Regarding the query on the "One-Pot Condensation Approach for the Synthesis of Some 1,8-Dioxo-octahydroxanthenes and 14-Aryl-14H-dibenzo[a,j]Xanthenes Using Lactic Acid as an Efficient and Eco-Friendly Catalyst," lactic acid serves as a homogeneous, eco-friendly acid catalyst alternative to solid sulfonated catalysts (20). It promotes the condensation reaction similarly by providing acidic conditions but with added benefits of biodegradability and lower environmental impact (21). Thus, while the selected text focuses on solid sulfonated catalysts, lactic acid represents a green catalyst option in one-pot condensation methods for synthesizing xanthenes, achieving efficient ring formation under mild, environmentally friendly conditions (22). The selected text explains that in the synthesis of xanthenes, solid sulfonated catalysts facilitate the acid-catalyzed condensation of aldehydes with phenolic compounds under mild conditions, efficiently forming the xanthene ring system (23). Their solid form offers advantages by preventing corrosion and simplifying purification. Regarding the query on the "One-Pot Condensation Approach for the Synthesis of Some 1,8-Dioxo-octahydroxanthenes and 14-Aryl-14H-dibenzo[a,j]Xanthenes Using Lactic Acid as an Efficient and Eco-Friendly Catalyst," lactic acid serves as a homogeneous, eco-friendly acid catalyst alternative to solid sulfonated catalysts. It promotes the condensation reaction similarly by providing acidic conditions but with added benefits of biodegradability and lower environmental impact (24). Thus, while the selected text focuses on solid sulfonated catalysts, lactic acid represents a green catalyst option in one-pot condensation methods for synthesizing xanthenes, achieving efficient ring formation under mild, environmentally friendly conditions. Would you like assistance in drafting a comparison of catalyst types for your synthesis section or help refining the discussion on lactic acid's role in this reaction. (25-27) Thus, the current effort focuses on the efficient and environmentally friendly synthesis of heterocyclic xanthene derivatives employing reusable solid sulfonated catalysts. (28-30)

Materials and Methods

1. Materials

All compounds, including solvents, dimedone, β-naphthol, and aromatic aldehydes, were purchased from commercial vendors and utilised without additional purification.

2. Preparation of Solid Sulfonated Catalyst

Using chlorosulfonic acid, a suitable support material was sulfonated to create the solid sulfonated catalyst. Strongly acidic $-SO_3H$ functional groups are affixed to the surface of the resultant catalyst.

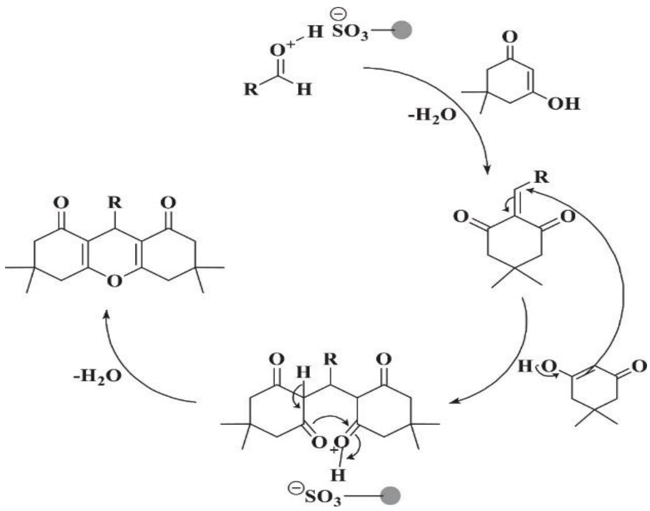
The catalyst was described by means of:

1. FT-IR spectroscopy
2. SEM: scanning electron microscopy
3. TGA, or thermogravimetric analysis

These methods verified the catalyst's structural stability and the effective addition of sulfonic acid groups.

3. General Procedure for Synthesis of Xanthene Derivatives

A mixture of dimedone (2 mmol), aromatic aldehyde (1 mmol), and a catalytic quantity of solid sulfonated catalyst was agitated at a high temperature without the use of a solvent. Thin layer chromatography (TLC) was used to track the development of the reaction. Following completion, the liquid was cooled, and filtration was used to separate the catalyst. Pure xanthene derivatives were obtained by recrystallising the crude product from ethanol.



1. Reaction Mechanism Sequence

Step 1: Activation of Aldehyde

The solid acid catalyst ($R-SO_3H$) protonates the carbonyl oxygen of the aromatic aldehyde, increasing the electrophilicity of the carbonyl carbon.

Step 2: Knoevenagel Condensation

Dimedone exists in its enol form. The active methylene carbon of one dimedone molecule attacks the activated carbonyl carbon of the aldehyde. This is followed by the elimination of a water molecule to form a **benzylidene dimedone intermediate**.

Step 3: Michael Addition
A second molecule of dimedone (acting as a Michael donor) attacks the activated double bond of the benzylidene intermediate (Michael acceptor). This forms a **Michael adduct** (bis-dimedone intermediate).

Step 4: Intramolecular Cyclization
The hydroxyl group of the Michael adduct attacks the carbonyl carbon of the other dimedone moiety, resulting in the formation of a **tricyclic xanthene ring system**.

Step 5: Dehydration
Final dehydration (loss of a second water molecule) from the cyclized intermediate, catalyzed by the solid acid, produces the stable **1,8-dioxo-octahydroxanthene derivative**.

2. General Reaction Scheme

1. **Reactants:** Aromatic aldehyde + 2 equivalents of dimedone (5,5-dimethyl-1,3-cyclohexanedione).
2. **Catalysts:** Sulfonated carbon, Silica-SO_3H, Sulfonated magnetic nanoparticles, or other Brønsted acidic materials.
3. **Conditions:** Often solvent-free, or in Ethanol/Water, typically heated
4. shows a comparison between the methods reported in the articles and the method performed in this work.
5. A comparison of the methods used in the articles and the method used in this work.

Entry	Catalyst	Conditions	Reaction time (min)	Yields%	Ref.
1	PEG	H_2O/reflux	~ 180	~ 90	(20)
2	activated zinc metal and solid NH_4C	Solvent free /MW	~ 50	~ 90	(21)
3	1-butyl-3-methylimidazoliumtetrafluoroborate (BMIF)	solvent free/~200 °C	~ 60	~ 90	(22)
4	Fe nanoparticles loaded in zeolite X (Fe-X)	Solvent-free, 90°C	~ 40	~ 90	(23)
5	$SO_3H@Fe_3O_4$ magnetic nanocatalyst	Solvent-free, 80 °C	~ 20	~ 90	(24)
6	$SO_3H@$ Bagasse	Solvent-Ethanol, 80°C	35-60	72-95	This work

4. Results and Discussion
For the synthesis of several xanthene derivatives, the designed catalytic system showed outstanding catalytic performance.
Benefits of the Catalyst
1. A high level of catalysis
2. Quick response time
3. Outstanding yield of products (85–95%)
4. A straightforward work-up process
5. Up to four or five cycles of reusability

According to studies, sulfonated heterogeneous catalysts greatly improve xanthene synthesis efficiency and enable solvent-free reactions, making the process safer and more environmentally friendly.
Different substituted aromatic aldehydes were successfully converted into the corresponding xanthene derivatives with high efficiency, demonstrating the versatility of the catalytic system.

5. Advantages of Solid Sulfonated Catalysts
Solid sulfonated catalysts have a number of benefits over traditional homogeneous acid catalysts.
1. Simple extraction from reaction mixtures
2. Recyclable and reusable
3. Less pollutants in the environment
4. Excellent thermal stability
5. Enhanced efficiency and selectivity

   They are promising catalysts for sustainable organic synthesis because of these qualities.

Conclusion
Using reusable solid sulfonated catalysts, an effective and eco-friendly process for the synthesis of heterocyclic xanthene derivatives has been devised in this work. High yields and quick reaction times are produced by the catalyst's efficient promotion of cyclisation and condensation processes. The catalyst's potential for environmentally friendly and sustainable organic synthesis is highlighted by its capacity to be recovered and reused repeatedly. This method offers a useful substitute for traditional homogenous acid catalysts in the synthesis of important heterocyclic compounds.

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Conflicts of interest
The authors declare that there are no conflicts of interest regarding the publication of this paper.

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