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# Water-Mediated Green Synthesis of Benzopyrans via a One-Pot Approach

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**Abstract:** *A convenient, efficient, and environmentally benign method has been developed for the synthesis of polyfunctionalized benzopyran derivatives using tricaprylylmethylammonium chloride (Aliquat-336) as a phase-transfer catalyst. The reaction proceeds efficiently in the presence of 10 mol% of Aliquat-336, providing a simple and effective approach for the preparation of a diverse range of benzopyran derivatives. The developed protocol offers several notable advantages, including good to excellent product yields, operational simplicity, mild reaction conditions, reduced environmental impact, and a straightforward work-up procedure. The use of Aliquat-336 as an effective catalyst demonstrates the potential of this methodology as a practical and greener alternative for the synthesis of biologically and synthetically important benzopyran frameworks.*

**Key Words:** Benzopyran derivatives; Aliquat-336; tricaprylylmethylammonium chloride; phase-transfer catalysis; one-pot synthesis; green chemistry; aqueous synthesis.

## I. INTRODUCTION

The concept of an ideal synthesis has become an important guiding principle in modern organic chemistry. An ideal synthetic process should ideally transform readily available, inexpensive, and structurally simple starting materials into the desired target molecule in a single operation, with maximum atom economy, high selectivity, minimal waste generation, and little or no environmental burden.[1] Such principles have gained considerable significance with the growing awareness of the environmental consequences associated with conventional chemical synthesis. The increasing generation of chemical waste, extensive use of volatile and hazardous organic solvents, consumption of large quantities of energy, and application of toxic reagents have encouraged researchers to develop cleaner and more sustainable synthetic methodologies.

Green chemistry has therefore emerged as an essential component of contemporary organic synthesis. The development of environmentally benign chemical processes aims to minimize or eliminate the use and generation of hazardous substances while maintaining high synthetic efficiency.[2] In particular, the replacement of toxic organic solvents, reduction of reaction steps, use of catalytic rather than stoichiometric reagents, improvement of atom economy, and development of simple purification procedures have become important objectives in synthetic organic chemistry. These requirements are particularly relevant to the pharmaceutical and fine-chemical industries, where large-scale chemical production can generate considerable quantities of waste and may involve hazardous reagents and solvents. Consequently, the design of economical, efficient, and environmentally acceptable synthetic methodologies has become a significant area of research in modern organic chemistry.

Among the different approaches developed to achieve these objectives, one-pot and multicomponent reactions (MCRs) have attracted considerable attention. A multicomponent reaction is a synthetic process in which three or more starting materials participate in a single reaction operation to produce a structurally complex product.[3] Such reactions provide an attractive alternative to conventional multistep synthesis because several bonds can be formed sequentially or simultaneously without isolation of intermediate products. Consequently, MCRs can substantially reduce reaction time, solvent consumption, energy requirements, purification steps, and overall waste generation.

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The one-pot approach is particularly valuable for the construction of heterocyclic compounds because structurally diverse and highly functionalized heterocycles can often be obtained from simple and commercially available starting materials. The ability to generate molecular complexity from relatively simple precursors in a single operation makes multicomponent reactions an important tool for both medicinal and synthetic chemistry.[4] Moreover, catalytic one-pot processes that avoid stoichiometric toxic reagents and excessive quantities of solvents are consistent with several fundamental principles of green chemistry.

Table 1. Major advantages of one-pot and multicomponent reactions

| Feature | Conventional multistep synthesis | One-pot/multicomponent synthesis |
|---|---|---|
| Number of reaction operations | Usually multiple | Generally one |
| Isolation of intermediates | Often required | Usually avoided |
| Solvent consumption | Relatively high | Reduced |
| Purification steps | Multiple | Generally fewer |
| Reaction time | Often longer | Usually shorter |
| Waste generation | Comparatively high | Lower |
| Energy consumption | May be high | Potentially reduced |
| Molecular complexity | Built stepwise | Can be generated rapidly |
| Operational simplicity | Moderate to complex | Generally simple |
| Green chemistry potential | Variable | High |

## II. BENZOPYRANS AS IMPORTANT HETEROCYCLIC SCAFFOLDS

Heterocyclic compounds occupy a central position in organic chemistry because of their extensive occurrence in natural products and their broad range of biological, pharmaceutical, and industrial applications. Among the numerous oxygen-containing heterocycles, benzopyrans represent an important class of compounds. Benzopyran frameworks occur as structural components of a variety of naturally occurring compounds and are widely distributed throughout the plant kingdom.[5] Their characteristic fused benzene–oxygen-containing heterocyclic framework provides considerable structural diversity and chemical versatility.

Benzopyran derivatives have attracted significant attention because of their applications in several areas of science and technology. Certain members of this class have been investigated for use as fragrances, food and cosmetic additives, biodegradable agrochemical candidates, optical brightening agents, fluorescent materials, and components of tunable dye-laser systems.[6–9] The diverse physicochemical properties of benzopyran derivatives, together with the possibility of introducing different functional groups into their molecular framework, make them valuable targets in synthetic organic chemistry.

More importantly, many benzopyran derivatives exhibit significant biological and pharmacological activities. Reported activities associated with benzopyran-containing compounds include anticoagulant, antianaphylactic, anticancer, spasmolytic, diuretic, antibacterial, antifungal, and insecticidal properties.[10] The biological importance of this heterocyclic framework has consequently stimulated extensive efforts toward the development of efficient methods for the synthesis of structurally diverse and polyfunctionalized benzopyran derivatives.

Table 2. Selected applications and biological importance of benzopyran derivatives

| Area | Reported significance |
|---|---|
| Natural products | Structural unit in numerous naturally occurring compounds |
| Pharmaceutical research | Valuable heterocyclic scaffold for bioactive molecules |
| Anticancer research | Several derivatives have demonstrated anticancer potential |
| Antibacterial activity | Reported for a number of substituted derivatives |
| Antifungal activity | Certain benzopyrans exhibit antifungal properties |
| Anticoagulant activity | Some members possess anticoagulant properties |

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| | |
|---|---|
| Agrochemicals | Potential biodegradable agrochemical applications |
| Food and cosmetics | Certain derivatives have applications as additives/fragrance materials |
| Optical materials | Used/investigated as fluorescent and optical materials |
| Dye chemistry | Potential application in tunable dye-laser systems |

Synthetic Approaches toward Polyfunctionalized Benzopyrans

The synthesis of polyfunctionalized benzopyrans has therefore become an attractive research area. Several synthetic strategies have been developed for constructing these heterocyclic systems. Among them, three-component one-pot condensation reactions provide an especially convenient approach because they allow the rapid assembly of the benzopyran framework from simple precursors.

A commonly employed strategy involves the condensation of ethyl cyanoacetate, an aldehyde, and an activated phenol under basic or amine-mediated catalytic conditions.[11] In this approach, the three reaction components undergo a sequence of condensation, nucleophilic addition, and cyclization processes to generate the corresponding polyfunctionalized benzopyran derivative. The methodology is synthetically attractive because different aldehydes and phenolic substrates can potentially be employed, thereby allowing structural diversification of the products.

However, despite the availability of several reported procedures, conventional approaches may suffer from certain limitations. These include moderate or low product yields in some cases, prolonged reaction times, use of environmentally undesirable solvents, requirement of relatively large quantities of catalyst or reagent, difficult product isolation, and generation of considerable amounts of chemical waste. In addition, the use of hazardous or expensive reagents can reduce the practical and environmental attractiveness of a synthetic protocol.

Table 3. General limitations associated with conventional benzopyran synthesis

| Limitation | Consequence |
|---|---|
| Use of hazardous organic solvents | Increased environmental and health concerns |
| Stoichiometric reagents | Increased waste generation |
| Multiple reaction steps | Increased operational complexity |
| Moderate yields | Reduced synthetic efficiency |
| Long reaction times | Increased energy and process costs |
| Difficult work-up | Additional solvent and purification requirements |
| Chromatographic purification | Increased waste and processing time |
| Expensive catalysts/reagents | Reduced economic feasibility |
| Poor substrate tolerance in some methods | Limited structural diversity |

Aliquat-336 as a Phase-Transfer Catalyst

Phase-transfer catalysis (PTC) represents an important strategy for facilitating reactions between reagents located in different phases, particularly when one reactant is present in an aqueous or ionic phase and another is present in an organic phase. Phase-transfer catalysts enhance the transport of reactive ionic species between immiscible phases, thereby increasing the rate and efficiency of chemical transformations.

Among the commercially available phase-transfer catalysts, tricaprylylmethylammonium chloride, commonly known as Aliquat-336, has received considerable attention because of its effectiveness, commercial availability, operational simplicity, and versatility in organic synthesis. Aliquat-336 is a quaternary ammonium salt capable of facilitating the transfer of reactive anionic species across phase boundaries. This property can enhance contact between otherwise poorly miscible reactants and consequently promote a variety of organic transformations.

The use of Aliquat-336 is particularly attractive from a practical perspective because it is readily available and can be employed in relatively small catalytic quantities. Its application can simplify reaction procedures and, under suitable conditions, reduce the requirement for conventional organic-solvent-based reaction systems. Thus, the development of catalytic methodologies based on Aliquat-336 is consistent with the broader objective of developing economical and environmentally more acceptable synthetic processes.

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Table 4. Desirable characteristics of Aliquat-336 in organic synthesis

| Property | Significance |
|---|---|
| Phase-transfer catalytic activity | Facilitates reactions between different phases |
| Commercial availability | Easily accessible for laboratory applications |
| Catalytic efficiency | Can be used in relatively low quantities |
| Operational simplicity | Easy incorporation into reaction procedures |
| Versatility | Applicable to a variety of organic transformations |
| Potential for greener processing | May reduce dependence on conventional reaction conditions |
| Cost effectiveness | Attractive for practical synthetic applications |
| Compatibility with one-pot reactions | Suitable for multicomponent synthetic strategies |

**Present Study**

Although phase-transfer catalysts have been successfully employed in numerous organic transformations, comparatively limited attention has been devoted to the application of Aliquat-336 as a catalyst for the synthesis of polyfunctionalized benzopyran derivatives. This observation prompted us to investigate the potential of Aliquat-336 for developing a simple, efficient, and environmentally more acceptable synthetic protocol for these biologically and synthetically important heterocyclic compounds.

In the present study, we report a convenient one-pot three-component synthesis of polyfunctionalized benzopyran derivatives using 10 mol% Aliquat-336 as a catalyst. The methodology is designed to provide an efficient route from readily available starting materials while minimizing operational complexity and purification requirements. The catalytic system promotes the formation of the desired benzopyran framework through a multicomponent condensation process and provides the corresponding products in good to excellent yields.

The developed method offers several practical advantages, including simple reaction conditions, catalytic use of Aliquat-336, good to excellent yields, operational convenience, reduced environmental impact, and a straightforward work-up procedure. Furthermore, the one-pot nature of the transformation eliminates the need for isolation of reaction intermediates, thereby reducing the number of synthetic operations and minimizing material and solvent consumption.

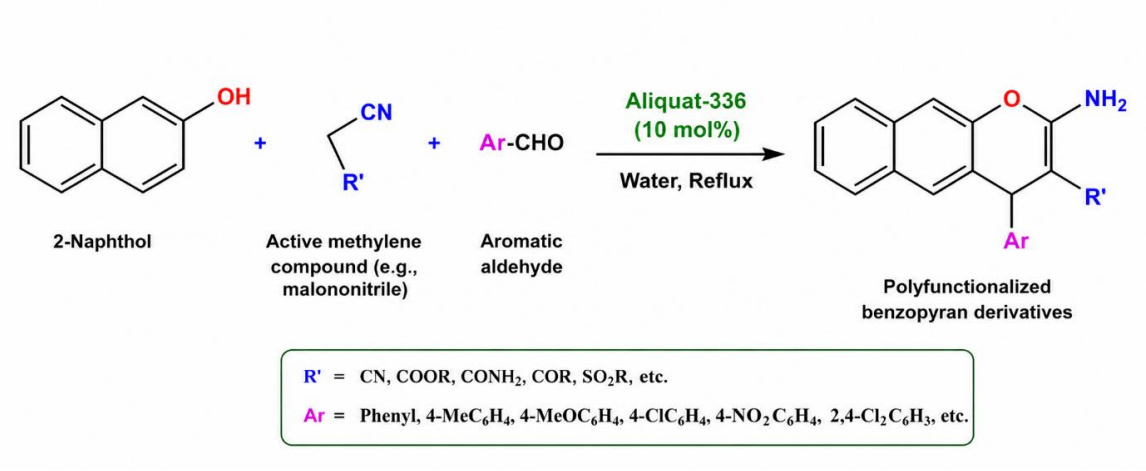
The present investigation therefore demonstrates the potential of Aliquat-336 as an effective catalyst for the preparation of polyfunctionalized benzopyran derivatives and provides a useful contribution toward the development of simpler and more environmentally acceptable methodologies in heterocyclic synthesis.

Table 5. Salient features of the present synthetic methodology

| Parameter | Present methodology |
|---|---|
| Target compounds | Polyfunctionalized benzopyran derivatives |
| Reaction type | One-pot multicomponent condensation |
| Number of components | Three |
| Catalyst | Tricaprylylmethylammonium chloride (Aliquat-336) |
| Catalyst loading | 10 mol% |
| Synthetic approach | One-pot synthesis |
| Major objective | Efficient and environmentally benign synthesis |
| Product formation | Good to excellent yields |
| Work-up | Simple and convenient |
| Purification | Minimized compared with multistep procedures |
| Environmental advantage | Reduced solvent/reagent burden |
| Practical advantage | Simple, economical, and operationally convenient |

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## III. EXPERIMENTAL

**General Experimental Procedure for the Synthesis of Polyfunctionalized Benzopyran Derivatives**

Aromatic aldehydes (1 mmol), alkyl cyanoacetates (1 mmol), and 2-naphthol (1 mmol) were mixed in the presence of Aliquat-336 (10 mol%) and water as the reaction medium. The resulting reaction mixture was heated under reflux for 4 h with continuous stirring. The progress of the reaction was monitored periodically by thin-layer chromatography (TLC) using an appropriate solvent system.

After completion of the reaction, as indicated by TLC, the reaction mixture was allowed to cool to room temperature. The desired benzopyran derivative was then extracted with ethyl acetate. The organic layer was separated and washed thoroughly with water to remove water-soluble impurities and residual catalyst. The organic phase was subsequently dried over anhydrous sodium sulfate ($Na_2SO_4$) and filtered. The filtrate was concentrated under reduced pressure to obtain the crude product.

The resulting solid product was purified by recrystallization from 95% ethanol to afford the corresponding polyfunctionalized benzopyran derivative in good to excellent yield. The physical and spectroscopic characteristics of the synthesized compounds were recorded, and their structures were confirmed by infrared (IR) spectroscopy, ^1H NMR spectroscopy, and elemental analysis. The analytical data obtained were found to be consistent with the proposed structures of the synthesized benzopyran derivatives.[12]

**Simple Work-Up and Product Isolation**

The developed procedure provides a simple and convenient work-up, involving extraction with ethyl acetate, washing with water, drying over anhydrous sodium sulfate, concentration, and recrystallization. The avoidance of multiple purification steps and the use of catalytic amounts of Aliquat-336 make this protocol operationally simple and potentially advantageous from an environmental and practical standpoint.

| S. No. | Phenol | R[1] | Ar | Yield (%)[a] | m.p. (°C)[b] |
|---|---|---|---|---|---|
| 1 | 1-Naphthol | –CN | $C_6H_5$ | 90 | 205–207[12] |
| 2 | 1-Naphthol | –CN | 2-Cl-$C_6H_4$ | 87 | 235–237[12] |
| 3 | 1-Naphthol | –CN | 3-Cl-$C_6H_4$ | 89 | 220–222[12] |
| 4 | 1-Naphthol | –CN | 4-Cl-$C_6H_4$ | 93 | 229–230[11] |
| 5 | 1-Naphthol | –CN | 2,4-$Cl_2C_6H_3$ | 85 | 215–217[12] |
| 6 | 1-Naphthol | –CN | 3-$NO_2$-$C_6H_4$ | 82 | 216–218[11] |
| 7 | 1-Naphthol | –CN | 4-$NO_2$-$C_6H_4$ | 87 | 236–238[12] |
| 8 | 1-Naphthol | –CN | 4-OH-$C_6H_4$ | 90 | 245–247[11] |

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| | | | | | |
|---|---|---|---|---|---|
| 9 | 1-Naphthol | –CN | 4-$CH_3O$-$C_6H_4$ | 92 | 184–186[11] |
| 10 | 2-Naphthol | –CN | $C_6H_5$ | 92 | 274–276[12] |
| 11 | 2-Naphthol | –CN | 4-$CH_3$-$C_6H_4$ | 77 | 255–257[12] |
| 12 | 2-Naphthol | –CN | 4-$NO_2$-$C_6H_4$ | 81 | 187–189[12] |
| 13 | 2-Naphthol | –CN | 4-$CH_3O$-$C_6H_4$ | 83 | 191–193[11] |
| 14 | 2-Naphthol | –CN | 2-$CH_3O$-$C_6H_4$ | 81 | 115–117[12] |
| 15 | 2-Naphthol | –CN | 2-Cl-$C_6H_4$ | 79 | 260–262[11] |
| 16 | 2-Naphthol | –COOEt | $C_6H_5$ | 93 | 165–167 |
| 17 | 2-Naphthol | –COOEt | 4-F-$C_6H_4$ | 86 | 237–239 |
| 18 | 2-Naphthol | –COOEt | 2-$NO_2$-$C_6H_4$ | 87 | 248–250 |
| 19 | 2-Naphthol | –COOEt | 4-$CH_3O$-$C_6H_4$ | 88 | 213–215 |
| 20 | 2-Naphthol | –COOEt | 2-Cl-$C_6H_4$ | 89 | 246–248 |

## IV. RESULTS AND DISCUSSION

The synthesis of the benzopyran derivatives was investigated through a one-pot three-component cyclocondensation reaction involving an active methylene compound, an aromatic aldehyde, and an activated phenol in water. The reaction mixture was heated under reflux in the presence of Aliquat-336 (10 mol%) as a phase-transfer catalyst. Upon completion of the reaction, the resulting benzopyran derivatives were isolated by a simple extraction procedure and purified by recrystallization from a suitable solvent.

The results obtained are summarized in Table 1. The developed protocol proved to be efficient for the preparation of a variety of polyfunctionalized benzopyran derivatives. It was observed that the nature and position of the substituents on the aromatic aldehyde had no significant adverse effect on the reaction outcome. Both electron-donating and electron-withdrawing substituents were well tolerated under the optimized reaction conditions, and the corresponding benzopyran derivatives were obtained in good to excellent yields. In particular, aromatic aldehydes bearing electron-withdrawing groups such as chloro and nitro substituents reacted smoothly to afford the desired products in satisfactory yields.

The nature of the phenolic component also influenced the product formation to some extent; however, both 1-naphthol and 2-naphthol participated effectively in the reaction. The observed substrate tolerance demonstrates the versatility of the present catalytic system for the synthesis of structurally diverse benzopyran derivatives.

**Effect of Catalyst Loading**

The amount of Aliquat-336 was found to play a crucial role in determining both the reaction rate and product yield. To establish the optimum catalyst concentration, the reaction was examined using different amounts of Aliquat-336 ranging from 1 to 10 mol%. A gradual increase in catalyst loading resulted in a corresponding improvement in the reaction rate and isolated yield, with the yield increasing from approximately 60% to 90% as the catalyst loading was increased.

The best results were obtained with 10 mol% of Aliquat-336, which provided efficient conversion of the starting materials and afforded the desired benzopyran derivatives in high yields. Interestingly, increasing the catalyst loading beyond 10 mol% did not result in any significant improvement in either reaction rate or product yield. Therefore, 10 mol% Aliquat-336 was selected as the optimum catalyst loading for the present study.

**Control Experiment**

To further establish the importance of the catalyst, a control experiment was performed under otherwise identical reaction conditions but in the absence of Aliquat-336. The reaction proceeded very slowly and afforded the desired benzopyran product in a poor yield of less than 30%. This observation clearly demonstrates the important role of Aliquat-336 in promoting the three-component cyclocondensation reaction.

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**Green Chemistry Aspect**

The use of water as the reaction medium represents an additional attractive feature of the present methodology. Water is inexpensive, readily available, non-flammable, and environmentally benign compared with many conventional organic solvents. Combining an aqueous reaction medium with a catalytic amount of Aliquat-336 and a one-pot multicomponent strategy provides a relatively simple and environmentally preferable approach to benzopyran synthesis.

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