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Sustainable One-Pot Synthesis of Polyhydroquinoline Derivatives Using [TMPSA][HSO4] in Aqueous Media

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Abstract: *A one-pot multicomponent condensation of aromatic aldehydes, dimedone, β-keto esters, and ammonium acetate has been developed for the efficient synthesis of polyhydroquinoline derivatives using* ***[TMPSA][HSO$_4$]*** *as a catalyst in aqueous medium 80^{o}C. Various solvents and catalyst loadings were systematically investigated to optimize the reaction conditions. Under the optimized conditions, the desired polyhydroquinoline derivatives were obtained in excellent yields ranging from* ***84–92%.*** *The structures of the synthesized compounds were confirmed by appropriate spectral analysis. The present protocol offers several advantages such as the use of an environmentally benign catalyst, good atom economy, operational simplicity, low cost, and easy work-up, making it an efficient and sustainable approach for the synthesis of polyhydroquinoline derivatives..*

Keywords: [TMPSA] [HSO$_4$], aromatic aldehydes, β-keto esters, ethanol, reflux

I. INTRODUCTION

Multi-component reactions have originated as efficient and impressive strategies in current synthetic organic chemistry offering the fantastic formation of numerous new bonds in a one-pot reaction. It also provides significant advantages over conventional linear type synthesis as having extraordinary atom economy, ease of implementation, and broad application character. Contrary for multi-step synthetic processes, numerous reactions and purification steps are involved which violates the most essential principles for the efficiency and practicability of the process. Due to that research in academia and industry has highly emphasized the use of MCRs for a comprehensive range of products.[1] The Hantzsch reaction is classified as a one-pot, three and four -component process for the synthesis of 1,4-dihydropyridine (DHP). Knoevenagel and Fries described an alteration of Hantzsch dihydropyridine synthesis. They conveyed the synthesis of unsymmetrical pyridine compounds by reacting various β-ketoesters.[2] In Knoevenagel and Fries modification, a variety of substituted β-ketoesters and 1,3-dicarbonyl compounds were used for the synthesis of unsymmetrical 1,4-DHP compounds. Among those 1,4-DHP compounds, substituted polyhydroquinoline derivatives have much more attention of chemists because of substantial biological activity.[3-4]

The synthesis of polyhydroquinoline derivatives (PHQ) is of great interest due to their biological properties such as nifedipine are calcium channel antagonists.[5] 2, 4-disubstituted PHQs exhibit potential as lipid modulating and anti-hyperglycemic agents.[6] Polyhydroquinoline is a vital structural design in the formulation of the drugs for the treatment of cardiovascular diseases, including hypertension. These exhibit biological properties such as antibacterial, antifungal, antituberculasis, antimalarial.[7]

Recently, much attention has been conferred towards the synthesis of polyhydroquinoline compounds owing to their diverse therapeutic and pharmacological properties. PHQ derivatives exhibit assets such as antiartherosclerotic, antitumor, vasodilator, bronchodilator, hepatoprotective activity, and geroprotective.[8] Polyhydroquinolines have also

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been shown to decrease cellular tau levels and hence hold potential in the exploration for therapies for Alzheimer's disease. [9]

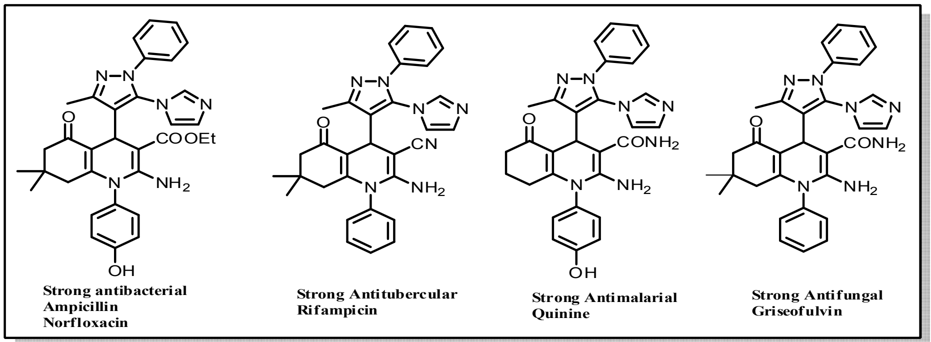
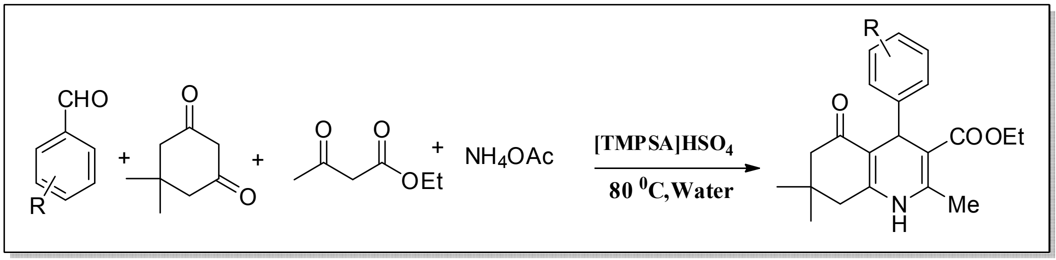


Fig. 1 Drug Scaffolds comprising of PHQ nucleus

Several PHQ derivatives correspondingly show activity against multidrug resistance proteins.[10] PHQs have been precisely applied as reducing agents for the direct reductive amination of aldehydes and ketones[11] as well as their oxidation to quinolines being also extensively studied.[12-13] Quioniline and its derivatives are the N containing heterocycles found in a broad range of natural products, together with alkaloids. These compounds fit in to this structural class have also been applied as dyes and intermediates in organic synthesis,[14-17] ligands for the formulation of OLED phosphorescent complexes[18] and the scheming of nano and mesostructures with better electronic and photonic belongings.[19-20]

Recognizing the significance of polyhydroquinoline derivatives in the production of several drugs, numerous protocols were reported. The most common route to polyhydroquinolines involves a reformed Hantzsch reaction comprising the multi-component coupling of an aldehyde with a β ketoester, a diketone, and ammonium acetate. The synthetic methodologies stated involving the catalyst such as ceric ammonium nitrate,[21] Baker's yeast,[22] Metal triflates,[23] zeolites,[24] organocatalysts,[25] sulphamic acid,[26] thioura dioxide,[27], thiazolium ion,[28] triphenyl phosphine,[29] GaCl$_3$,[30] $Al_2(SO_4)_3$,[31] I_2,[32] metal oxide NPs.[33] PHQs syntheses have been reported under catalyst-free conditions, but the approaches grieve with longer reaction times.[34] Solvent and catalyst-free procedures comprising grinding the reaction mixtures have also been described.[35] However these techniques need the use of solvent for product isolation and are not always responsive. PHQs have also been produced using microwave irradiation.[36] The enantioselective synthesis PHQs have been proposed using an organocatalyst derived from phosphoric acid.[37]

In spite of the above efforts to progress the polyhydroquinolines synthesis, a number of shortcomings have been noticed like low yield, extended reaction time, tedious workup practice and perilous solvents. Acidic task-specific ionic liquids (TSILs), which own the expedient features of solid acids and mineral acids, are intended to substitute conventional mineral liquid acids, such as sulfuric acid and hydrochloric acid. Indeed, the practice of Brønsted-acidic TSILs as catalysts is an expanse of ongoing research, though, development and exploration of acidic TSILs are presently in the initial phase. The N,N,N-trimethyl-N-propanesulfonic acid ammonium hydrogen sulfate [TMPSA] [HSO$_4$] is a cheap, novel and halogen free -SO$_3$H functional ionic liquid that bear an alkane sulphonic acid group in acyclic trimethylammmonium cation. [TMPSA] [HSO$_4$] was used as catalyst in many organic transformations such as Pechmann condensation.[38] Considering these lacunas, herein we have schemed a clean and modest protocol for the synthesis of polyhydroquinoline-3-carboxylate and its derivatives by using ionic liquid N,N,N-trimethyl-N-propanesulfonic acid ammonium hydrogen sulfate [TMPSA] [HSO$_4$] as a green catalyst in aqueous media at 80 ^{0}C.



Scheme 1: Synthesis of polyhydroquinoline derivatives using [TMPSA] [HSO4]

II. EXPERIMENTAL

All the required chemicals were bought from Merck, SD fine and solvents for chromatography were obtained from Qualigens, Spectrochem were distilled before use. Reactions were monitored by thin layer chromatography. All the synthesized PHQ derivatives were inveterate by the assessment of their spectroscopic data 1H NMR, 13C NMR, IR, and Mass. 1H NMR were documented at room temperature on Advance Bruker 400 MHz spectrometer (NCL, Pune) in CDCl3 employing tetramethylsilane as an internal standard and IR spectra were obtained with a Perkin Elmer Spectrum RX FTIR ((NCL, Pune)) instrument using KBr Pellets in the range of 400-4000 Cm-1. Mass spectra were observed from Micromass QTOF liquid chromatography mass spectroscopy. Melting points were measured in open glass capillary tube and are uncorrected. Yields refer to isolated pure products.

General Procedure of polyhydroquinoline synthesis

A mixture of aryl/ heteryl aldehyde (1 mmol), ethyl acetoacetate (1 mmol), dimedone (1 mmol), ammonium acetate (2 mmol) was taken in round bottom flak with 15 ml of water. To this reaction mixture 5 mol% of N,N,N-trimethyl-N-propanesulfonic acid ammonium hydrogen sulfate [TMPSA] [HSO4] was added. The reaction mixture was heated to 80 0C with constant stirring for appropriate time till the reaction completed (monitored by TLC using 8:2 system of pet-ether:ethyl acetate). The reaction mixture was cooled to room temperature and filtered to isolate the crude product, washed with water and recrystallized with ethanol-water to acquire pure product. The pure product was isolated by column chromatography using silica gel (mesh 60-120) and a mixture of pet-ether and ethyl acetate (8:2) as mobile phase. Structures of pure compounds were substantiated using ^{1}HNMR, IR, Mass spectrometry and melting points with authentic samples and literature data. The filtrate was distilled at 100 0C to remove water leaving behind [TMPSA] [HSO4], recovered catalyst was washed with ethyl acetate, dried at ambient temperature and used for the next run.

III RESULT AND DISCUSSION

As a feature of our enduring research about the use of Bronsted acid ionic liquids in organic transformations, and in the formulation of novel protocol for unsymmetrical polyhydroquinoline derivatives, we have executed the use of [TMPSA] [HSO4] for the synthesis of ethyl-4-aryl or heteryl-hexahydro-trimehtyl-5-oxoquinoline-3-carboxylates. The optimization for the reaction was determined using classical reaction that entailed of benzaldehyde, dimedone, ethyl acetoacetate and ammonium acetate. Initially the reaction of benzaldehyde, dimedone, ethyl acetoacetate, and ammonium acetate was carried out without solvent and catalyst at reflux condition. However, the reaction was sluggish and corresponding polyhydroquinoline was obtained in 6-7 h and yield was very poor. The same reaction was then carried out using [TMPSA] [HSO4] as Bronsted acid catalyst in aqueous medium at room temperature affording the corresponding PHQ in good yield in 2.2 h. The influence of temperature on the reaction was studied (table 1). The yield of the product 5a was significantly affected by reaction temperature. The development of product at low temperature does not favor even after experience to long reaction time. We got that the reaction time decreases and yield of product get increases with increase in temperature. It may perhaps because of rise in temperature advance the liberation of ammonia from ammonium acetate and improves the condensation.

Table 1: Effect of temperature on the synthesis of 5a PHQ derivative

Entry	**Reaction Temperature (^{0}C)**	**Time (h)**	**Yield^b (%)**
1	R.T.	2.2	65
2	50	1.9	74
3	70	1.5	85
4	80	1.2	88

^{a}Reaction condition: benzaldehyde (1 mmol), ethyl acetoacetate (1 mmol), dimedone (1 mmol), ammonium acetate (2 mmol), and [TMPSA] [HSO_4] catalyst (5 mol %) in water (15 ml) at different temperature, ^{b}Yields of the isolated products.

To optimize the catalyst amount in the above stated reaction, we have done a model study with benzaldehyde with dimedone, ethyl acetoacetate and ammonium acetate using varying amount of catalyst such as 1 mol%, 2 mol%, 3 mol%, 4 mol% and 5 mol% of catalyst at 80^{o}C in water solvent. The observations depicted in the table 2 clearly exhibits that in the absence of the catalyst, product was obtained in low yield after 1.5 h., while pleasing results were perceived in the presence of 5 mol% of ionic liquid catalyst at 80 0C in terms of reaction time and yield obtained.

Table 2: Effect of catalyst amount on the synthesis of 5a PHQ derivative

Entry	**Catalyst amount (mol %)**	**Time (h)**	**Yield^b (%)**
1	No Catalyst	7.0	60
2	1	2.5	68
3	2	2.2	74
4	3	1.9	80
5	4	1.4	83
6	5	1.2	88
7	6	1.0	88

^{a}Reaction condition: benzaldehyde (1 mmol), dimedone (1 mmol), ethyl acetoacetate (1mmol), ammonium acetate (2 mmol), at different amount of [TMPSA] [HSO4] in aqueous medium (15 ml) at 80 ^{0}C,^{b}Yields of the isolated products.

Furthermore, we have demonstrated the effect of various solvents from non-polar to polar on catalytic activity. As a model reaction, the reaction of benzaldehyde with dimedone, ethyl acetoacetate and ammonium acetate catalyzed by 5 mol% ionic liquid in various solvents is summarized in table 3. The several solvent (Polar and Non-polar) offered the product in the lower to moderate yield (70-88 %, table 3), excellent yield obtained in water (92 %).

Table 3: Solvent effect on the Synthesis of 5a PHQ derivative

Entry	**Solvent**	**Time (h)**	**Yield^b (%)**
1	Hexane	2.5	70
2	CCl_4	2.6	80
3	THF	2.2	78
4	DMF	1.8	81
5	CH_3CN	1.7	82
6	$CHCl_3$	2.2	80
7	Water	1.2	88
8	Ethanol	1.5	88
9	Toluene	2.1	74

[a]Reaction condition: benzaldehyde (1mmol), ethyl acetoacetate (1 mmol), dimedone (1 mmol), ammonium acetate (2 mmol), in various solvents at 80 ^{0}C using 5 mol% catalyst,
[b]Yields of the isolated products.

From table 3, it is confirmed that water is clearly the excellent solvent for these reactions because water is benign, cheap, and safe rivalled with other organic solvents.

The reusability of catalyst is one the preeminent crucial features of green chemistry. The employed room temperature ionic liquid [TMPSA] [HSO4] can be reused in the synthesis of polyhydroquinoline derivatives easily. After completion of reaction, the catalyst [TEBSA] [HSO4] was separated and reused continuously for four times deprived of any major loss of product yields and competence of catalyst as mentioned in table 4.

Table 4: Reusability of catalyst for the synthesis of 5a PHQ derivative

Entry	Yield ^{b} (%)	Time (h)	Cycle
1	88	1.2	I
2	84	1.3	II
3	81	1.6	III
4	78	1.8	IV
5	70	2.2	V

[a]Reaction condition: Benzaldehyde (1 mmol), dimedone (1 mmol), ethyl acetoacetate (1 mmol), ammonium acetate (2 mmol) and 5 mol % [TMPSA] [HSO_4] in water (15 ml) at 80^{0}C, [b]Yields of the isolated products.

To analyze the synthetic horizon, efficacy and generality of this protocol, several instances elucidating this process for the synthesis those polyhydroquinoline derivatives were studied. To demonstrate the general applicability of this scheme, various substituted aromatic aldehydes and heteroaromatic aldehydes were proficiently reacted with dimedone, ethyl acetoacetate and ammonium acetate in the same ideal conditions i.e. in water at 80 0C. The outcomes are summarized in table 5. The influence of electron and the nature of substituents on the aromatic ring did show expected strong effects in terms of yields and reaction time under these reaction conditions. Aromatic aldehydes bearing electron withdrawing groups such as nitro, chloro, or electron-releasing groups like hydroxy, N,N dimethyl amino, alkoxy were used and they were found to react fine to produce the corresponding polyhydroquinoline in good to excellent yields. Aromatic aldehydes bearing electron-withdrawing groups on the aromatic ring (table 5, entries 2, 4, and 9) react faster than electron-donating groups (table 5, entries 5-8 and10-11).

Heteroaromatic aldehydes (table 5, entries 12-14) required extended reaction times, which is mostly due to the electron-releasing and steric effects of the heteroatoms.

Table 5: Synthesis of polyhydroquinoline-3-carboxylate derivatives using [TMPSA] [HSO_4] as catalyst in aqueous media

Entry	Aldehyde	Product	Time (h)	Yield^b (%)	Melting Point (^{0}C)
1		**5a**	1.2	88	204-206
2		**5b**	1.3	86	208-210
3		**5c**	1.2	86	200-202

Entry	Aldehyde	Product	Time (h)	Yield^{b} (%)	Melting Point (^{0}C)
4		**5d**	1.1	88	242-244
5		**5e**	1.4	82	254-256
6		**5f**	1.3	82	260-262
7		**5g**	1.5	84	230-232
8		**5h**	1.4	81	204-206
9		**5i**	1.5	85	202-204
10		**5j**	1.5	88	214-216
11		**5k**	1.6	85	234-236

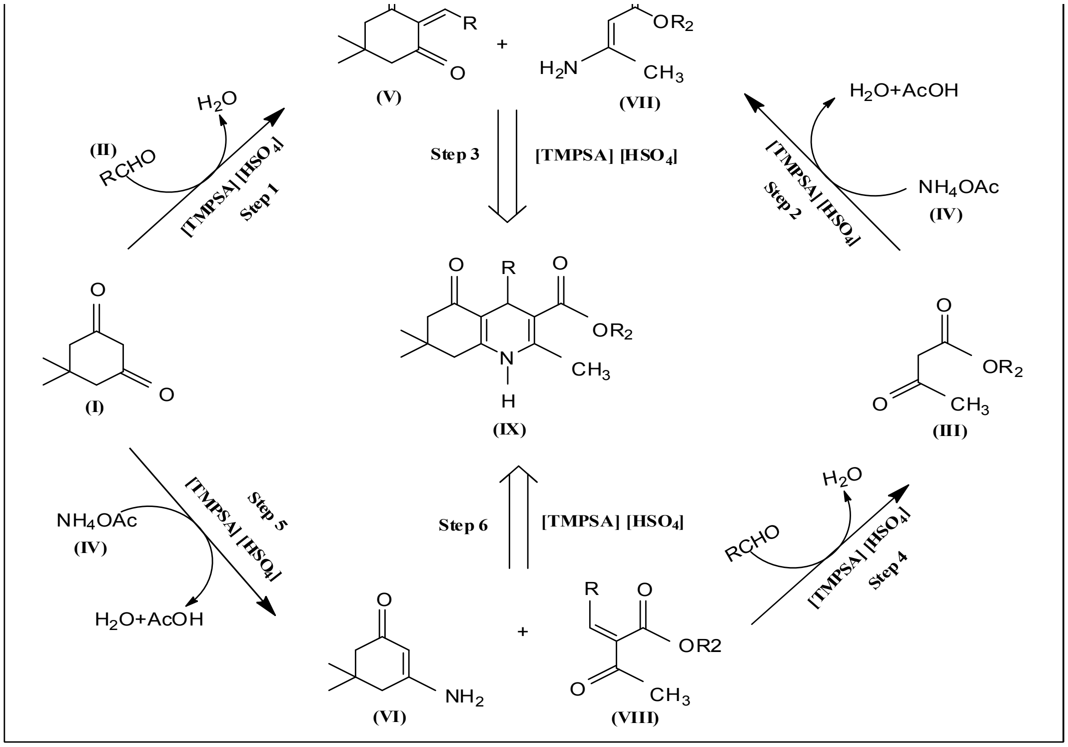
Entry	Aldehyde	Product	Time (h)	Yield^b (%)	Melting Point (^{0}C)
12		**5l**	1.8	86	230-232
13		**5m**	1.9	86	244-246
14		**5n**	1.9	88	224-226

^{a}Reaction condition: substituted aldehyde (1 mmol), ethyl acetoacetate (1 mmol), dimedone (1 mmol), ammonium acetate (2 mmol), in aqueous media at 80 ^{0}C using 5 Mol% catalyst,

^{b}Yields of the isolated products.

Plausible Mechanism

The Bronsted acidity of [TMPSA] [HSO4] catalyzed the reaction via forming H-bonding with the carbonyl groups of aldehyde. The dimedone (I) or β-ketoester (III) reacted with aldehyde (II) via Knoevenagel condensation to form intermediate (V) or (VI). The β-ketoester (III) reacted with ammonium acetate to yield intermediate (VII) or (VIII). Both these intermediates undergo cyclization to generate desired product (IX).



Scheme 2

IV. CONCLUSION

In conclusion, we have depicted a modest and highly competent method for one pot four component synthesis of polyhydroquinoline derivatives by condensing aldehydes, ethyl acetoacetate, dimedone and ammonium acetate in presence of catalytic amount of ionic liquid [TMPSA] [HSO4] at 80 0C condition in aqueous media. The catalyst has been recovered and reused for five cycles without much loss in it's catalytic activity. The developed protocol has the advantages such as simple & mild experimental procedure, use of readily available inexpensive and reusable catalyst, use of aqueous medium for carrying out reactions, allowing high yields for various aromatic and heteroaromatic aldehydes, more environment friendly method making it a best substitute to existing approaches.

V. ACKNOWLEDGMENT

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