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PEG-SO_3H as a catalyst in aqueous media: A simple, proficient and green approach for the synthesis of quinoline derivatives

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Abstract. A convenient and efficient method was developed for the synthesis of quinolines, an important class of potentially bioactive compounds. The quinoline derivatives were prepared in water, an excellent solvent in terms of environmental impact and with reduced waste production. PEG-SO_3H effectively catalysed the one-pot synthesis of quinolines by the condensation of *o*-aminoaryl ketones and carbonyl compound with high yields (75–95%). The compounds were isolated by simple filtration in a high purity form.

Keywords. Poly (ethylene glycol)-bound sulphonic acid; quinoline; aqueous media; green chemistry.

1. Introduction

Catalysis lies at the heart of countless chemical protocols from academic research laboratories to the chemical industry. A variety of products, such as medicines, fine chemicals, polymers, fibres, fuels, paints, lubricants, and a myriad of other value added products essential to humans, would not be feasible in the absence of catalysts.[1] In recent years, there has been a rapid growth in the development of novel polymer-supported compounds such as supported catalysts, reagents and scavengers. These species allow rapid and simplified procedures and their use has become widespread in solution-phase organic synthesis and combinatorial chemistry.[2] PEG-SO_3H is one of the examples of modified PEG, which has been reported to be an efficient reagent or catalyst for the synthesis of organic compounds such as the synthesis of 3,4-dihydropyrimidones,[3] thiocyano-hydrines,[4] triazolo[1, 2-a]indazole-triones[5] and Beckmann rearrangement.[6]

Solvents are chemical substances used in huge amounts for many different applications. In many cases, organic solvents are chemical substances derived from petrol, and have a negative impact on the health and the environment. One of the key areas of Green Chemistry is the elimination of solvents in chemical processes or the replacement of hazardous solvents with environmentally benign solvents. The use of water as a medium for organic reactions is one of the latest challenges for modern organic chemists. Water emerged as a useful alternative solvent for several organic reactions owing to many of its potential advantages such as safety, economy and environmental concern.[7,8]

The synthesis of quinolines has been of considerable interest to chemists because their oxygen heterocycles may contribute to potential antimalarial, antibacterial, antiasthmatic, antihypertensive, antiinflammatory, antiplatelet and tyro kinase PDGF-RTK inhibiting properties.[9–16]

For the synthesis of quinolines, various methods have been reported including the Skraup,[17] Conrad–Limpach–Knorr,[18,19] Pfitzinger,[20,21] Friedländer,[22,23] and Combes.[24,25]

However, the Friedlander condensation is still considered as a popular method for the synthesis of quinoline derivatives.[26–35] In this method, *o*-amino benzophenone condenses with ketones or β-diketones to yield quinolines. Nevertheless the development of new synthetic methods for the efficient preparation of heterocycles containing quinoline fragment is therefore an interesting challenge. Therefore, in this report, we describe the synthesis of quinoline derivatives by treatment of 2-aminobenzophenone with various carbonyl compounds using PEG-SO_3H as new catalysts in water with high yields (75–95%).

2. Experimental

2.1 *Materials and apparatus*

Carbonyl compounds and *o*-aminobenzophenone were purchased from Merck Chemical Companies. Purity determinations of the products were accomplished by

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TLC on silica-gel polygram SILG/UV 254 plates. Melting points were determined in electrothermal 9100 system open capillaries and were uncorrected. IR spectra were taken on a Perkin Elmer 781 spectrometer in KBr pellets and reported in cm^{-1}. NMR spectra were measured on a Bruker DPX 250 MHz spectrometer in DMSO-d_6 with chemical shift given in ppm relative to TMS as internal standard.

2.2 *Preparation of PEG-SO_3H*

To a magnetically stirred mixture of 6.00 g (1 mmol) of Poly(ethylene glycol) (PEG-6000) in 10 mL of dichloromethane, 0.67 mL of chlorosulphonic acid (10 mmol) was added drop-wise at 0°C during 1 h. HCl gas was removed from the reaction vessel immediately and the mixture was stirred for 12 h. The mixture was filtered and washed with 20 mL of diethylether and dried at room temperature to afford 5.10 g of PEG-SO_3H as a white powder. The number of H^+ sites on the PEG–SO_3H was determined by acid–base titration to be 0.5 meq/g.

2.3 *General procedure for the preparation of quinoline derivatives*

To a mixture of carbonyl compounds (1.0 mmol) and 2-amino-5-chlorobenzophenone or 2-aminobenzophenone (1.0 mmol) was added PEG-SO_3H (0.2 g) in H_2O (3 mL). The mixture was stirred at 60°C for appropriated reaction time (table 3). The progress of the reaction was monitored by TLC. After completion of the reaction, the precipitated solid was filtered and washed with water. The crude solid product was crystallized from EtOH to afford the pure quinoline product in high purity in excellent yield. Structural assignments of the products are based on their ^{1}H NMR, ^{13}C NMR, MS and IR spectra.

2.3a *Compound (**3c**): 7-chloro-9-phenyl-3,4-dihydro-1-2H-acridinone*: Yellow solid, m.p 184°C (Lit [41] 185°C), IR (KBr, cm^{-1}) ν_{max} 3024, 2975, 2870, 1698, 1549, 1476, 1380, 1210, 1075, 1007, 970, 838, 697. ^{1}H NMR (400 MHz, $CDCl_3$)δ 2.26 (q, 2H, J = 6.4 Hz), 2.72 (t, 2H, J = 6.4 Hz), 3.37 (t, 2H, J = 6.4 Hz), 7.17 (t, 2H), 7.42 (s, 1H), 7.53 (m, 3H), 7.69 (d, 1H, J = 8.8 Hz), 8.01 (d, 1H, J = 8.8 Hz), MS (m/z, %): 308((M +2)−1, 34), 306 (M −1, 100), 281(5), 280 (29), 278(15), 253(4), 244(10), 215(27), 188(12) , 153(17), 120(20), 107(15).

2.3b *Compound (**3i**): methyl-6-chloro-2-methyl-4-phenyl-3-quinolinecarboxylat*: Yellow solid, m.p 135°C, (Lit [41] 135°C), IR (KBr, cm^{-1}): υ: 3035, 2958, 2900, 1749, 1561, 1455, 1402, 1297, 1237, 1182, 1070, 872, 767. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.74 (s, 3H), 3.56 (s, 3H), 7.25–8.01 (c, 8H). ^{13}C NMR (62.9 MHz, $CDCl_3$) δ 23.7, 52.2, 125.2, 125.8, 128.0, 128.5, 128.8, 129.1, 130.5, 131.2, 132.4, 134.9, 145.5, 146.1, 145.9, 154.9, 168.6. MS(m/z, %): 313(M +2, 31), 311(M^+, 100), 296(6), 281(50), 279(97), 254(14), 251(16), 236(4), 211(10), 189(34), 175(52), 108(37), 94(33), 74(17).

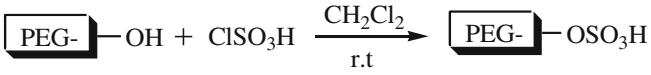
2.3c *Compound (**3x**): 2-Chloro-11-phenyl-7,8,9,10-tetrahydro-6H-cyclohepta[b]quinoline*: Yellow solid, m.p 195°C, (Lit [42] 195°C), IR (KBr, cm^{-1}): υ: 3080, 3050, 2930, 2850, 1615, 1600, 1500, 1470, 1360, 1180, 990, 870, 820, 680, ^{1}H NMR (400 MHz, $CDCl_3$): δ: 1.60 (s, 2H), 1.84 (s, 4H), 2.68 (c, 2H) 3.26 (c, 2H), 7.22–7.96 (c, 8H).^{13}C NMR (62.9 MHz, $CDCl_3$) δ26.9, 28.4, 30.7, 31.8, 40.1, 125.1, 127.7, 127.9, 128.6, 129.0, 129.3, 130.3, 131.3, 134.8, 136.9, 144.2, 144.7, 165.1, MS (m/z, %): 308((M+2)−1, 33), 306(M-1, 100), 292(9), 280 (15), 277(12), 252(13), 242(17), 228(18), 215(18) ,201(10), 188(10), 127(23), 120(25), 107(15).

2.3d *Compound (**3ee**): 6-Chloro-2,4-diphenylquinoline*: Yellow solid mp: 206°C, (Lit. [42] 208°C), IR (KBr, cm^{-1}): υ: 3019, 2985, 1580, 1508, 1465, 1355, 1150, 1005, 840, 790, 755. ^{1}H NMR (400 MHz, $CDCl_3$): δ: 7.25–8.19 (c, 14H),^{13}C NMR (62.9 MHz, $CDCl_3$) δ 120.5, 124.5, 126.5, 127.5, 128.7, 128.8, 128.9, 129.4, 129.6, 130.4, 131.7, 132.2, 137.5, 139.2, 147.2, 148.4, 157.1, MS(m/z, %): 316(M +2, 43), 314(M^+, 100), 280(27), 277(18), 250(6), 236(7), 201(17), 175(13), 139(57), 125(19).

3. Results and discussion

The supported acidic PEG catalyst was prepared via anchoring chlorosulphonic acid onto polyethylene glycol. This polymeric catalyst was used as an efficient Brönsted acid for different organic functional group transformations either as reagent or as catalyst under heterogeneous and homogenous. The catalyst can easily be prepared from the readily available reagents, chlorosulphonic acid and poly ethylene glycol to give PEG modified sulphuric acid (scheme 1).

In order to evaluate the catalytic efficiency of PEG–SO_3H and to determine the most appropriate reaction conditions, initially a model study was carried out on

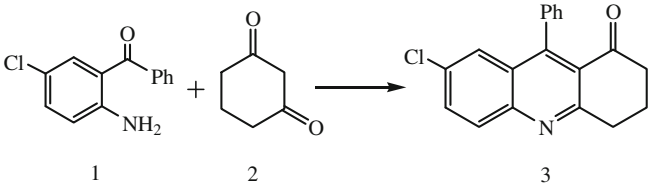


Scheme 1. Preparation of PEG-SO_3H.

the synthesis of quinoline 3 (scheme 2) by the condensation of 2-amino-5-chlorobenzophenone 1 and 1,3-cyclohexadione 2 in different sets of reaction conditions. In preliminary experiment, this reaction was carried out in various solvents, with PEG-SO_3H (0.2 g or 0.1 mmol H^+) as a catalyst, in 60°C for 15 min. The reaction proceeded perfectly in polar solvents (table 1, entries 1, 2, 10, 11), but the yields decreased when the reaction was carried out in non-polar solvents (table 1, entries 8, 9, 12). It was very surprising that the reaction proceeded in excellent yields (92%) in aqueous medium (table 1, entry 1).

To obtain the optimized reaction conditions, we also changed temperature and the amount of catalyst. The results are summarized in table 2. Consequently, among the tested temperature and the amount of catalyst, the condensation of 2-amino-5-chlorobenzophenone and 1,3-cyclohexadione was best catalysed by 0.2 g of PEG-SO_3H in water at 60°C. Control experiments indicate that in the absence of the catalyst, the reaction at the same condition gives quinoline in a rather low yield of 25% (table 2, entry 1).

To establish the generality and applicability of this method, 2-amino-5-chlorobenzophenone/2-aminobenzophenone and carbonyl compounds were subjected to the same reaction condition to furnish the corresponding quinolines in good to excellent yields (table 3, scheme 3). Not only diketones (table 3, entries 1–11) but also ketones (table 3, entries 12–17) afforded the desired products in good to excellent yields (76–90%) in short reaction time (40–75 min) under given condition. It is delighted that the reaction time of 1,3-diphenyl propane-1,3-dione was longer than those of acetyl acetone, which is probably due to low reactivity of carbonyl groups. Also, the reaction time of 2-aminobenzophenone and dicarbonyl compounds was longer than those of 2-amino-5-chlorobenzophenone. This behaviour could result from the more reactivity of 2-amino-5-chlorobenzophenone by electron-withdrawing groups (Cl). The reaction of cyclic diketones took place faster than open chain analogues.



Scheme 2. A selected reaction model for the synthesis of quinoline.

Table 1. The effect of various solvent in the reaction 2-amino-5-chlorobenzophenone (1 mmol), 1,3- cyclohexadione (1 mmol), catalyst (0.2 g), solvent (3 mL), stirred at 60°C for 15 min.

Entry	Solvent	Yield%	Entry	Solvent	Yield%
1	H_2O	92	8	*n*-Hexane	Trace
2	EtOH	80	9	$CHCl_3$	Trace
3	CH_3CN	60	10	DMSO	70
4	THF	40	11	MeOH	85
5	CH_2Cl_2	40	12	Dioxane	Trace
6	Toluene	20	13	DMF	20
7	EtOAC	75	14	Solvent free	76

These reactions also proceeded with acetophenone derivatives (table 3). In these cases the reaction times are longer. It may be due to the less activity of acetophenone derivatives than dicarbonyl compounds. All the aforementioned reactions (table 3) delivered good product yields and accommodated a wide range of aromatic carbonyl compound bearing both electron-donating and electron-withdrawing substituents. The reactivity of different aromatic carbonyl compounds was influenced by the nature and position of the substituents on the aromatic ring. The aromatic carbonyl derivatives having an electron-donating substituent were highly reactive and gave the products in excellent yields (entries 19, 20, 24, 25). When the aromatic carbonyl compounds containing electron-withdrawing group were used, the reaction time was longer (entries 23, 26, 29). In all cases, the obtained product was isolated by a simple filtration, washed with water and purified by recrystallization from ethanol.

In comparison with other catalysts employed for the synthesis of quinoline from *o*-amino benzophenone and carbonyl compound, PEG-SO_3H showed a much higher

Table 2. Effect of temperature and the amount of catalyst on the synthesis of 2-quinoline derivatives via a condensation of 2-amino-5-chlorobenzophenone and 1,3-cyclohexadione in the presence of PEG-SO_3H in water.

Entry	Catalyst (g)	Yield%			
		r.t	60°C	80°C	120°C
1	None	0	10	15	25
2	0.05	30	40	55	65
3	0.1	50	55	75	70
4	0.15	70	78	80	70
5	0.2	77	92	96	85
6	0.3	88	94	88	78
7	0.5	87	76	70	65

Table 3. Reaction times and yields of quinolones 3 by the PEG-SO_3H-catalysed Friedlander reaction in water[a].

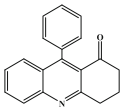
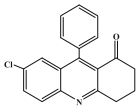
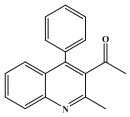
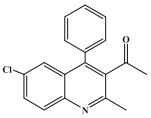
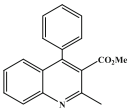
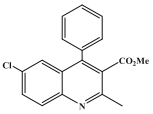
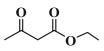
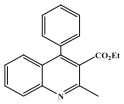
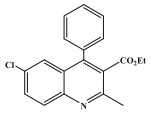
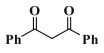
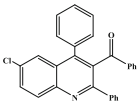
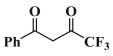
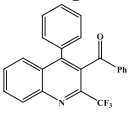
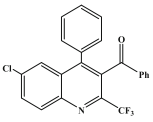
Entry	Substrate 1	Substrate 2	Quinoline 3	Reaction time (min)	Yield (%)[b]
1	1a 	2a 	3a 	15	90
2	1b 	2a	3c 	15	92
3	1a	2d 	3e 	25	92
4	1b	2d	3f 	20	95
5	1a	2g 	3h 	55	87
6	1b	2g	3i 	50	88
7	1a	2k 	3l 	40	88
8	1b	2k	3m 	35	89
9	1b	2n 	3o 	60	80
10	1a	2p 	3q 	50	75
11	1b	2p	3r 	45	77

Table 3. (continued).

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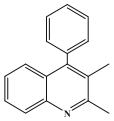
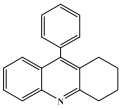
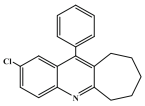
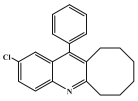
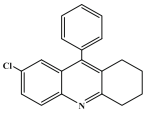
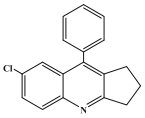
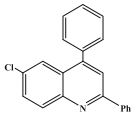
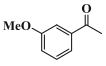
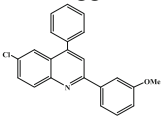
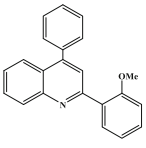
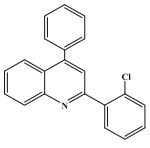
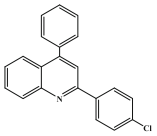
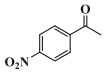
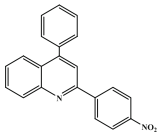
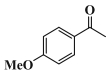
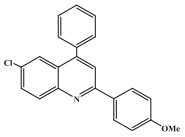
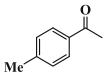
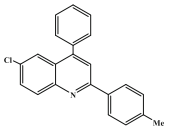
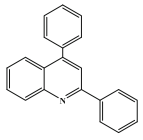
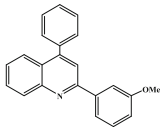
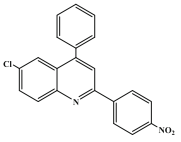
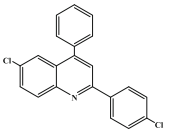
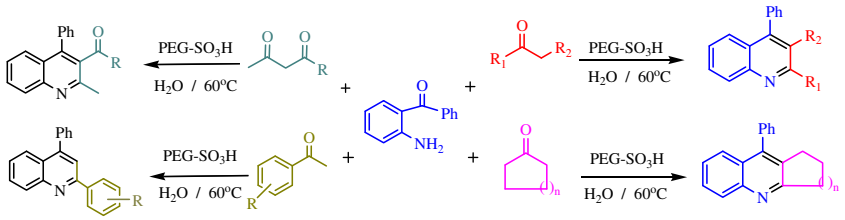
12	1a	2s 	3t 	75	76
13	1a	2u 	3v 	45	90
14	1b	2w 	3x 	40	87
15	1b	2y 	3z 	55	82
16	1b	2u	3aa 	50	87
17	1b	2bb 	3cc 	70	86
18	1b	2dd 	3ee 	40	87
19	1b	2ff 	3gg 	35	85
20	1a	2hh 	3ii 	35	85
21	1a	2kk 	3ll 	45	76

Table 3. (continued).

Entry	Substrate 1	Substrate 2	Quinoline 3	Reaction time (min)	Yield (%)[b]
22	1a	2mm 	3nn 	45	79
23	1a	2oo 	3pp 	50	82
24	1b	2qq 	3rr 	40	86
25	1b	2ss 	3tt 	50	85
26	1a	2uu 	3vv	50	79
27	1a	2dd	3ww 	55	84
28	1a	2ff	3xx 	40	82
29	1b	2oo	3yy 	50	81
30	1b	2mm	3zz 	40	82

[a]Reaction conditions: carbonyl compounds (1.0 mmol), 2-amino-5-chlorobenzophenone or 2-aminobenzophenone (1 mmol), PEG-SO_3H (0.1 mmol H^+) and H_2O (2 mL); reactions conducted at 60°C
[b]The yield refers to pure isolated product



Scheme 3. Reaction of 2-aminobenzophenone and various carbonyl compounds afforded the corresponding quinolines.

Table 4. One-pot condensation of *o*-amino benzophenone, carbonyl compound (a, b) in the presence of different catalysts.

Run	Catalyst	Condition	Solvent	Time(h)	Yield
1	PEG-SO_3H	60°C	H_2O	45(min)	90 (this work)[a]
2	$Ag_3PW_{12}O_{40}$	Reflux	EtOH	3.5	87 [36a][a]
3	HCl	100–200°C	H_2O	1	68 [36b][a]
4	$HClO_4/SiO_2$	Reflux	CH_3CN	3	92 [36c][a]
5	$Zr(HSO_4)_4$	Reflux	H_2O	13	87 [37a][a]
6	bmimCl-$ZnCl_2$	r.t	Ionic liquids	24	80 [38][a]
7	PEG-SO_3H	60°C	H_2O	40(min)	88 (this work) [b]
8	$Y(OTf)_3$	r.t	CH_3CN	6	83 [39][b]
9	$NaHSO_4/SiO_2$	70	None	4	80 [40a][b]
10	Amberlyst-15	Reflux	EtOH	2.5	87 [40b][b]
11	HCl	Microwave	HCl	6	60 [37b][b]

[a]cyclohexanone , [b]ethyl-3-oxobutanoate

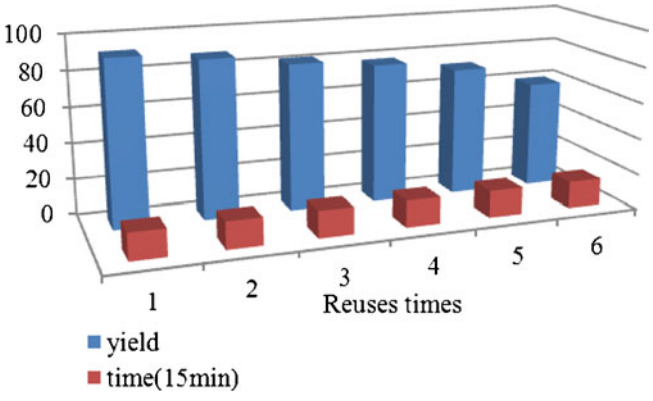


Figure 1. Reuses performance of the catalysts.

catalytic activity in terms of very shorter reaction time and mild conditions (table 4).

It is noteworthy to mention that the catalyst is recyclable and could be reused without significant loss of activity. It could be recovered by filtration of product, evaporation of solvent and washing with diethyl ether. The recycled catalyst could subject to a second or even another reaction. In the model reaction the results of the first experiment and the subsequent were almost consistent in yield after 6 runs (92%, 88%, 82%, 78%, 72% and 60%, figure 1).

4. Conclusion

In conclusion, an efficient synthesis of quinoline derivatives has been achieved by a one-pot coupling reaction of carbonyl compounds and *o*-aminobenzophenone using catalytic amounts of PEG–SO_3H in water. Simple reaction procedures, inexpensive catalysts and single product formation make this an attractive protocol over the existing procedures. This protocol offers flexibility in tuning the molecular complexity and diversity. Pure product was obtained, without using any chromatographic techniques, simply by recrystallization from ethanol.

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