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Highly efficacious preparation of 3,3′-(arylmethylene)-bis(2-hydroxynaphthoquinone) derivatives catalyzed by a nanorod-structured organic–inorganic hybrid material

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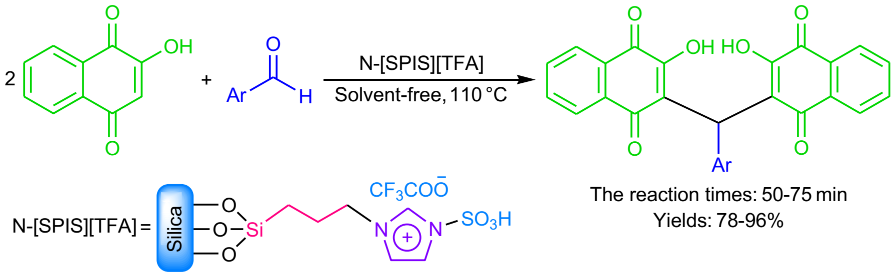
Abstract

A nanorod-structured organic–inorganic hybrid material namely nanorod-[SiO_2–Pr–Im–SO_3H][CF_3CO_2] (N-[SPIS][TFA]) has been applied as a highly efficacious, dual-functional, and recoverable catalyst for the solvent-free preparation of 3,3’-(arylmethylene)-bis(2-hydroxynaphthoquinone) {3,3′-(arylmethylene)-bis(2-hydroxynaphthalene-1,4-dione)} derivatives in high yields and relatively short times. The one-pot pseudo-multi-component reaction of 2-hydroxynaphthoquinone (2-hydroxynaphthalene-1,4-dione) (2 eq.) and arylaldehydes (1 eq.) has been used for the preparation of these compounds.

Graphic abstract



The reaction times: 50-75 min
Yields: 78-96%

Keywords 3,3′-(arylmethylene)-bis(2-hydroxynaphthoquinone) · Organic–inorganic hybrid material · Nanorod-[SiO_2–pr–im–SO_3H][CF_3CO_2]; (N-[SPIS][TFA]) · Pseudo-multi-component reaction · Solvent-free

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Introduction

Compounds bearing naphthoquinone moiety are of importance, since they have a wide range of biological, pharmaceutical, and industrial applications [1–14]. For example, they show antioxidant [1], antifungal [1], antiplatelet [2], antibacterial [3], antimalarial [4], anti-inflammatory [5], antithrombotic [6], anti-proliferative [7], antiviral [8], antitumor [9], CDC25 phosphatase inhibitor [10], anti-allergic [11], and antimicrobial [12] activities. Moreover, these compounds can be utilized as pigments [13, 14]. 3,3′-(Arylmethylene)-bis(2-hydroxynaphthoquinone) {3,3′-(arylmethylene)-bis(2-hydroxynaphthalene-1,4-dione)} derivatives are a kind of naphthoquinone-containing compounds, which can be prepared via the one-pot pseudo-multi-component reaction of 2-hydroxynaphthoquinone (2-hydroxynaphthalene-1,4-dione) (2 eq.) and arylaldehydes (1 eq.) in the presence of a catalyst [15–22]. It is worth noting that a few catalysts have been reported for the reaction of 2-hydroxynaphthoquinone (2 eq.) and arylaldehydes (1 eq.) leading to 3,3′-(arylmethylene)-bis(2-hydroxynaphthoquinone) derivatives; so, it is highly desirable to introduce new efficacious catalysts for this reaction.

Hybrid materials are composed from at least two different constituents wherein inorganic and organic constituents are put together by special interactions, and increase the synergistic of their functional properties [23]. They have attracted much attention in diverse scientific, industrial, and pharmaceutical fields [23–45]. Hybrid materials have been applied as electrode [24], sensor [25], antitumor [26], and platform for in vitro cell manipulation [27]. Furthermore, they have been used for decomposition of hazardous organic industrial pollutants in aqueous medium [28], energy storage [29], drug delivery [30], and bioimaging [30]. Organic–inorganic hybrid materials have been, especially, used as catalysts in organic synthesis [31–52]. Among the diverse inorganic compounds [31–55], silica is usually preferable to utilize as inorganic component for production of hybrid materials, due to possessing some unbeatable characteristics, consisting of safety, eco-friendly nature, appropriate thermal and chemical stability, low cost, non-corrosiveness, simple functionalization, having large surface-to-volume ratio (nanomaterials), and easy isolation from reaction medium [35–46].

Solvent-free conditions [56–61] and multi-component reactions [62–67] are practical, well-known, useful and green technique which has been used in organic synthesis; their advantages have been mentioned in the literature [56–67].

Considering the above topics, we have introduced a highly efficacious method for the preparation of 3,3′-(arylmethylene)-bis(2-hydroxynaphthoquinone) derivatives via the one-pot pseudo-three-component reaction of 2-hydroxynaphthoquinone (2 eq.) and arylaldehydes (1 eq.) using a nanorod-structured organic–inorganic hybrid catalyst namely nanorod-[SiO_2–Pr–Im–SO_3H][CF_3CO_2] (N-[SPIS][TFA]) under solvent-free conditions.

Experimental

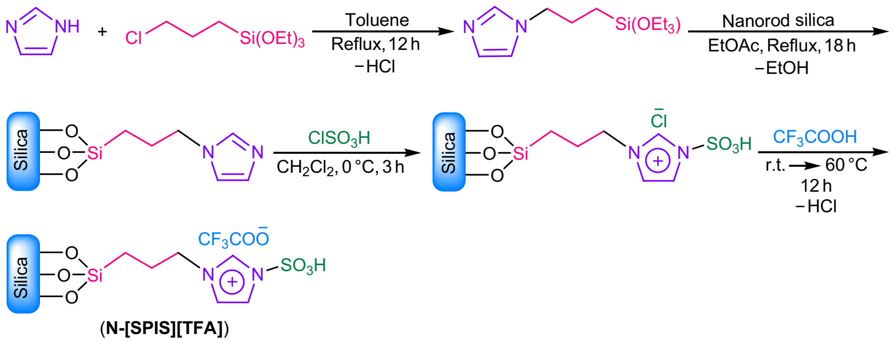
Materials and instruments

The reactants and solvents were bought from Fluka or Merck Chemical Companies. Nanorod-[SiO_2–Pr–Im–SO_3H][CF_3CO_2] (N-[SPIS][TFA]) was synthesized according to our recently published procedure in this Journal (Scheme 1) [35]. To identify the synthesized 3,3′-(arylmethylene)-bis(2-hydroxynaphthoquinone) derivatives, their melting points/spectral data were compared with those reported in the literature. Thin layer chromatography (TLC) (silica gel SIL G/UV 254 plates) was used for observation of the reactions progress. FT-IR was run on Bruker instrument (model: Tensor 27, Germany). ^1H and ^{13}C NMR were recorded on Bruker Avance DPX, FT-NMR spectrometer. Melting points were measured using a Thermo Scientific 9200 instrument in open capillary tubes. Mass spectra were obtained by a Shimadzu GC–MS-QP 1100 Ex device.

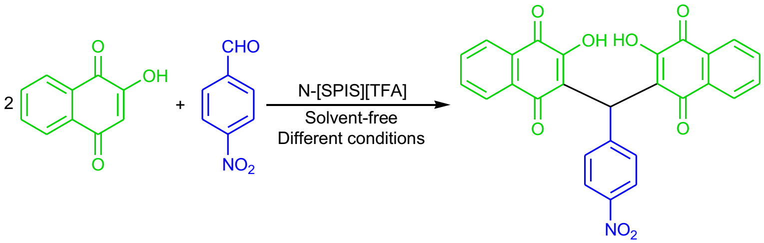
General procedure for the preparation of 3,3′-(arylmethylene)-bis(2-hydroxynaphthoquinone) derivatives

A mixture of 2-hydroxynaphthoquinone (2 mmol, 0.348 g), aldehyde (1 mmol for solid aldehydes; 1.1 mmol for liquid ones), and N-[SPIS][TFA] (0.020 g) was vigorously stirred by a small rod at 110 °C. After completing the reaction (as observed by TLC), EtOAc (30 mL) was added, stirred for 2 min under reflux conditions, followed by centrifugation and decanting to isolate the unsolvable catalyst (which was washed by EtOAc, dried and used for the next run). The EtOAc resulted from the decanting was evaporated, and the precipitate (crude product) was purified by recrystallization from EtOH/H_2O.

Note: Selected spectral data and original spectrums of the products have been presented in supplementary material.



Scheme 1 The synthesis of N-[SPIS][TFA]



Scheme 2 The model reaction

Table 1 The results on optimization of the reaction conditions (Scheme 2)

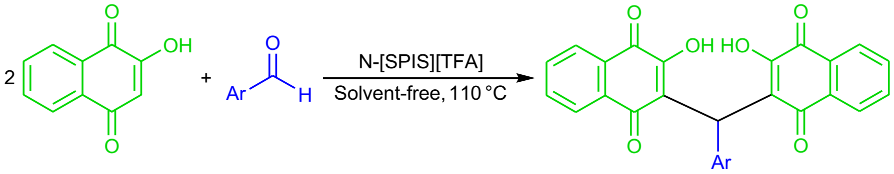
Entry	The catalyst amount (g)	Temp. (°C)	Time (min)	Yield (%)
1	–	110	50	17
2	0.015	110	50	81
3	0.020	110	50	93
4	0.025	110	45	93
5	0.020	100	50	79
6	0.020	115	50	93

Results and discussion

Initially, the reaction of 2-hydroxynaphthoquinone (2 mmol) and 4-nitrobenzaldehyde (1 mmol) was examined in the absence of catalyst and also using different amounts of nanorod-[SiO_2–Pr–Im–SO_3H][CF_3CO_2] (N-[SPIS][TFA]) at a range of 100–115 °C under solvent-free conditions (Scheme 2), in order to optimize the reaction conditions relative to the catalyst amount and temperature. The results are summarized in Table 1. Examining the reaction in the absence of catalyst at 110 °C afforded low yield of the desired 3,3′-(arylmethylene)-bis(2-hydroxynaphthoquinone) (Table 1, entry 1). The optimized conditions were attained when 0.020 g of the catalyst was applied at 110 °C; in these conditions, the desired 3,3′-(arylmethylene)-bis(2-hydroxynaphthoquinone) was produced in 93% after 50 min (Table 1, entry 3). Increasing the temperature up to 115 °C did not improve the reaction results (Table 1, entry 6). Increment of the catalyst amount up to 0.025 g slightly decreased the reaction time (Table 1, entry 4); nevertheless, 0.020 g was chosen as the optimal catalyst amount; because performing the reaction using fewer amount of the catalyst is more economical.

After acquiring the optimal conditions, various derivatives of 3,3′-(arylmethylene)-bis(2-hydroxynaphthoquinone) were prepared through the reaction of 2-hydroxynaphthoquinone (2 eq.) and different arylaldehydes (1 eq.); the results are reported in Table 2. As it can be observed in Table 2, all products were obtained in good to excellent yields and in relatively short times. When

Table 2 The solvent-free synthesis of 3,3′-(arylmethylene)-bis(2-hydroxynaphthoquinone) derivatives



Product No	Ar	Time (min)	Yield[a] (%)	M.p. (°C) [lit.]
1	C_6H_5	55	96	208–210 (202–204) [16]
2	4-ClC_6H_4	75	92	181–183 (180–182) [16]
3	4-FC_6H_4	75	83	174–176 (177–178) [20]
4	4-BrC_6H_4	75	96	196–198 (195–197) [20]
5	2,4-$Cl_2C_6H_3$	60	78	205–207 (200–202) [20]
6	3-$O_2NC_6H_4$	60	91	154–156 (151–152) [19]
7	4-$O_2NC_6H_4$	50	93	179–181 (177–178) [18]
8	3,4-$(CH_3O)_2C_6H_3$	75	84	178–180 (143–145) [19]
9	4-$CH_3OC_6H_4$	60	92	223–225 (220–222) [17]
10	4-$CH_3C_6H_4$	50	94	171–173 (170–172) [18]

[a] Isolated yield

benzaldehyde was used as aldehyde substrate, the related product was synthesized in excellent yield (Table 2, product **1**). Among the halogen-bearing aldehydes, 4-chloro and 4-bromobenzaldehyde gave high to excellent yields; however, 4-fluoro and 2,4-dichlorobenzaldehyde afforded good yields (Table 2, products **2**–**5**). When aldehydes containing electron-withdrawing substituents (3-nitro and 4-nitrobenzaldehyde) were applied in the reaction, the corresponding products were obtained in high yields (Table 2, products **6** and **7**). Among the aldehydes possessing electron-donating substituents, 4-methoxy and 4-methylbenzaldehyde gave the desired products in high yields; but, 3,4-dimethoxybenzaldehyde afforded the corresponding product in good yield (Table 2, products **8**–**10**). Considering the obtained results, it can be said that N-[SPIS][TFA] is a highly efficacious and general catalyst for the preparation of 3,3′-(arylmethylene)-bis(2-hydroxynaphthoquinone) derivatives.

Recoverability and reusability of N-[SPIS][TFA] were studied for the synthesis of 3,3′-(arylmethylene)-bis(2-hydroxynaphthoquinone) **1**. The catalyst was recovered as mentioned in the experimental section. It was reusable for two times with negligible decrement of the yield and increment of the reaction time (Fig. 1, runs 2 and 3). Nevertheless, the reaction yield was decreased to 85% in third recycling (Fig. 1, run 4).

N-[SPIS][TFA] can act as a dual-functional catalyst; SO_3H is an acidic group, and trifluoroacetate is a weak basic group [35]. Based on this theme and the literature [16, 20], a reasonable mechanism was proposed for the synthesis of

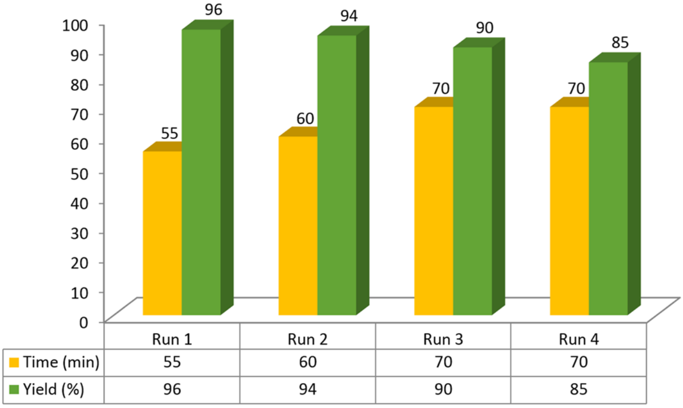


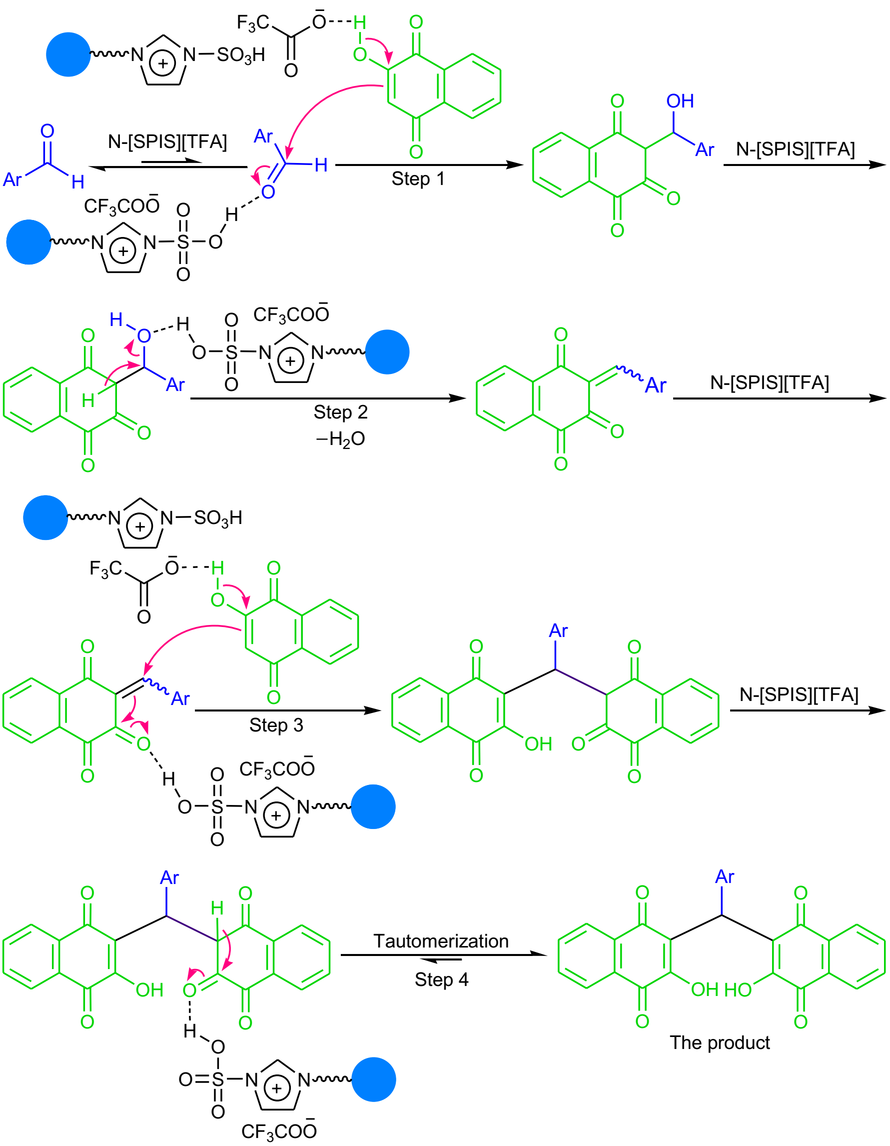
Fig. 1 The reusability results of N-[SPIS][TFA]

3,3′-(arylmethylene)-bis(2-hydroxynaphthoquinone) derivatives (Scheme 3). The catalyst can facilitate all steps of the reaction, its roles, which are clearly illustrated in the mechanism, consist of: (1) activation of the electrophiles by the SO_3H to react with the nucleophiles (Scheme 3, steps 1 and 3), (2) activating the nucleophiles by the trifluoroacetate anion to react with the electrophiles (Scheme 3, steps 1 and 3), (3) help for elimination of H_2O (Scheme 3, step 2), and (4) acceleration of tautomerization (Scheme 3, step 4).

In another study, the results and the reaction conditions of N-[SPIS][TFA] for the production of compound **1** were compared with those of the reported catalysts (Table 3). As Table 3 demonstrates, the reaction yield of N-[SPIS][TFA] was higher than the other catalysts, and the reaction time of our catalyst was shorter than some reported catalysts. Moreover, in our work, the reaction has been performed under solvent-free conditions. Nevertheless, the reaction temperature in our work is higher than the others.

Conclusions

Briefly, we have developed a new protocol for the preparation of 3,3′-(arylmethylene)-bis(2-hydroxynaphthoquinone) derivatives from 2-hydroxynaphthoquinone and arylaldehydes. The advantages of this protocol consist of efficacy, wide scope, high yields, relatively short reaction times, application of solvent-free conditions, and a nanorod-structured organic–inorganic hybrid catalyst (and consequently possessing the advantages of these two), studying a reaction that have been scarcely reported in the literature, simple purification of the products using recrystallization, recoverability of the catalyst and good agreement with green chemistry principles.



Scheme 3 The reaction mechanism

Table 3 The synthesis of compound 1 using N-[SPIS][TFA] and the reported catalysts

Catalyst	Conditions	Time (min)	Yield (%)	References
N-[SPIS][TFA]	Solvent-free, 110 °C	55	96	–
LiCl	H_2O, reflux	720	83	[16]
H_2SO_4	H_2O/EtOH, reflux	15	90	[17]
LiCl	H_2O, 25 °C, ultrasonic irradiation	5	91	[18]
LiCl	H_2O, 75 °C, microwave irradiation	15	87	[18]
$H_2O/[bmim][BF_4]$[a,b]	90 °C	240–360	85–92	[19]
Sulfamic acid	H_2O/EtOH, r.t	960	92	[20]
Lipase	EtOH, 60 °C	120	88	[21]
O-Carboxymethyl chitosan	H_2O/MeOH, 70 °C	50	89	[22]

[a] 1-Butyl-3-methylimidazolium tetrafluoroborate

[b] In this research, compound 1 has not been synthesized; thus, we tabulated the ranges of the reaction times and yields

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