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Brönsted acid hydrotrope combined catalysis in water: a green approach for the synthesis of indoloquinoxalines and bis-tetronic acids

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Abstract

The present work describes the applications of Brönsted acid hydrotrope combined catalyst (BAHC) as a mild, efficient and reusable catalyst for synthesis of indoloquinoxalines and bis-tetronic acids in water. Using BAHC, we synthesized many indoloquinoxaline derivatives from isatins and o-phenylene diamine using 10 mol% PTSA in 40% aqueous hydrotropic (NaPTS) solution at room temperature with 83–90% yields. On the other hand, the reaction of tetronic acid with the aldehydes/isatins forms bis-tetronic acids with 83–88% yields through Knoevengel condensation-Michael addition pathway in same BHAC. Moreover, the BAHC can be recycled upto 5th cycles with slight decrease in product yields. The extremely simple operational methodology, green solvent, ambient reaction conditions and high yields render this approach extremely appealing for the synthesis of different heterocyclic compounds.

Keywords Brönsted acid hydrotrope combined catalyst (BAHC) · Water · Indoloquinoxalines · Bis-tetronic acids

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Introduction

Environmental benign organic transformations have attracted much interest in organic chemistry by using renewable resources and reduction of waste materials. A streamline approach emerged out in this context is use of aqueous medium as a reaction solvent, which significantly provides platform for eco-friendly organic synthesis [1]. Though, the use of aqueous condition offers many advantages, the major problem associated with the use of water as a reaction medium is less solubility of organic reactants with decreased selectivity of reaction. To alleviate these drawbacks, several strategies like use of ultrasonication, use of co-solvents like DMF, DMSO and surfactants, supercritical fluids have been employed [2]. Among these strategies, use of hydrotropes (surface-active compounds), which was introduced by Carl Neuberg [3], exhibited more opportunity to solubilize many reactants in water through phenomenon called 'hydrotropy.' Hydrotropes enhance the solubility of many organic compounds in water up to 200 fold by associative mechanism [3, 4]. The salient features of hydrotrope include stack-like arrangement of molecules and amphiphilic molecular structure. The key use of hydrotropes as a reaction media depends on the nature of hydrotrope used and its minimum hydrotropic concentration (MHC). MHC is a concentration, above which there is maximum solubility of reactants in aqueous phase [5, 6]. Hydrotropes has attractive advantages such as it is non-toxic, inexpensive, and readily available. It has good solubilizing power and can be recyclable [7]. In addition, hydrotropes are also suitable under microwave and ultrasonic conditions [8, 9]. These promising properties of hydotropes make it a privileged reaction medium for various organic transformations such as multicomponent synthesis of pyrazole derivatives [10], the coupling reactions [11], regioselective synthesis of β-hydroxytriazoles [12] and diversity-oriented synthesis of quinazolinone derivatives [13].

The chemistry of heterocyclic compounds has remarkable importance in organic chemistry [14], as a wide range of heterocyclic compounds have different pharmacological properties and clinical applications [15–18]. In addition, some heterocycles find application in the field of material chemistry [19–24]. Heterocyclic biorelevant scaffolds such as quinoxalines and tetronic acids represent an interesting template in organic and medicinal chemistry (Fig. 1). Quinoxaline moiety is frequently found as core nucleus in natural products particularly in alkaloids, as well as in diverse bioactive compounds [25, 26]. Some derivatives of quinoxalines have significant applications in agrochemicals and material sciences [27–30]. Tetronic acids are a subclass of β-hydroxy butenolides that have received great attention in the synthetic chemistry [31–33], as well as they play an essential role in the field of medicinal chemistry, especially as antibiotic [34], HIV-1 protese inhibitors [35], anticoagulant [36], antifungal [37], antibacterial [38], analgesic [39], anti-inflammatory [40] and anticancer agent [41].

Considering the importance of all the above compounds and our previous work for the synthesis of quinoxalines and pyrido[2,3-*b*]pyrazines [42], we report herein a Brönsted acid hydrotrope combined catalyst (BAHC) for the synthesis of indoloquinoxaline and bis-tetronic acid derivatives in water at room temperature (Scheme 1).

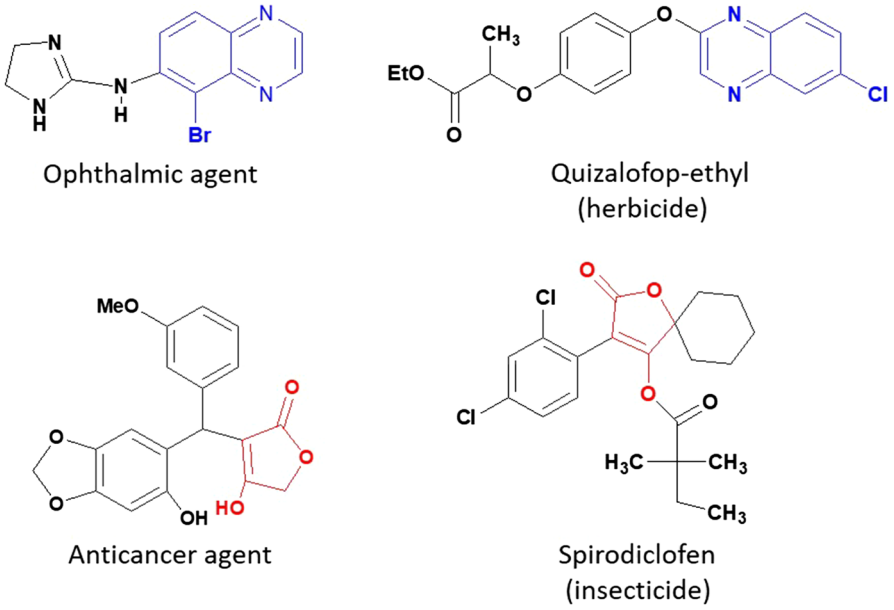
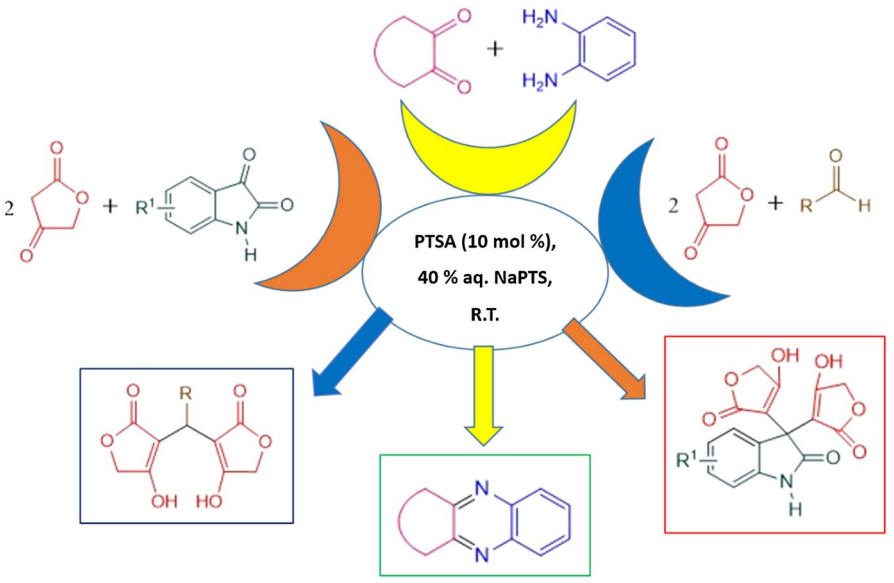


Fig. 1 Bioactive quinoxaline and tetronic acid scaffolds



Scheme 1 Synthesis of indoloquinoxalines and bis-tetronic acids using BAHC in water

Experimental

General

All the chemicals were obtained from Sigma Aldrich/Spectrochem and used without further purification. Sodium p-toluene sulphonate (NaPTS hydrotrope) was purchased from Alfa-Aesar company. TLC was carried out using silica gel G 60 F_{254} Plates (Merck). IR spectra were recorded on a Perkin–Elmer FT-IR 783 spectrophotometer. ^{1}H NMR, and ^{13}C NMR spectra were recorded on a Bruker AC (300 MHz for ^{1}H NMR and 75 MHz for ^{13}C NMR) spectrometer using $CDCl_3$ or DMSO-d_6 as solvent and tetramethylsilane (TMS) as an internal standard. Melting points were determined using DBK melting point apparatus and are uncorrected.

Typical procedure for the synthesis of indoloquinoxalines

Isatins/ninhydrine/acenaphthoquinone (1.0 mmol), o-phenylene diamine (1.0 mmol) and PTSA (10 mol %) were added in a round bottom flask, containing 40% aq NaPTS (5 mL) and the reaction mixture was stirred at room temperature. The reaction progress was monitored by thin-layer chromatography (TLC). After completion of the reaction, the mixture was diluted with water (20 mL). The filtrate was washed with water and dried affording the corresponding crude products, which on recrystallization using ethanol gave pure products.

Typical procedure for the synthesis of bis-tetronic acids

Tetronic acid (2.0 mmol), aldehydes/isatins (1.0 mmol) and PTSA (10 mol %) were added in a round bottom flask, containing 40% aq NaPTS (5 mL) and the resulting reaction mixture was stirred at room temperature. After completion of the reaction, as monitored by TLC, the mixture was diluted with water (20 mL). The filtrate was washed with water and dried affording the corresponding crude products, which on recrystallization using ethanol gave pure products.

Spectral data of representative compounds

Compound 3a

Solid; M.P.: 285–287 °C; IR (KBr): 3452 (N–H stretching), 3134, 3034, 2857, 1621 (C=N stretching), 1589 (C=C Stretching), 1414, 1342, 1262, 1206, 1124, 778, 730 cm^{-1}; ^{1}H-NMR (300 MHz, DMSO-d_6): δ 7.20–7.25 (t, 1H, $J=15$ Hz), 7.52–7.79 (m, 4H), 7.98–8.00 (d, 1H, $J=6$ Hz), 8.17–8.20 (d, 1H, $J=9$ Hz), 8.32–8.35 (d, 1H, $J=9$ Hz), 11.46 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ

111.88, 119.02, 120.66, 122.16, 125.50, 127.62, 128.31, 129.81, 131.21, 139.01, 140.68, 141.11, 144.39, 147.08 ppm.

Compound 5b

Solid; M.P.: 240–242 °C; IR (KBr): 3018, 1624 (C=N stretching), 1503 (C=C Stretching), 1434, 1302, 1240 (C–C stretching), 1086 (C–N stretching), 1018, 842, 764 cm^{-1}; ^{1}H-NMR (300 MHz, DMSO-d_6): δ 7.79–7.82 (dd, 2H, $J=3.6$ Hz, $J=6$ Hz), 7.90–7.95 (dd, 2H, $J=9$ Hz, $J=8.1$ Hz), 8.17–8.20 (dd, 2H, $J=3$ Hz, $J=6$ Hz), 8.32–8.35 (d, 2H, $J=6$ Hz), 8.43–8.46 (d, 2H, $J=7.2$ Hz); ^{13}C NMR (75 MHz, DMSO-d_6): δ 121.10, 128.41, 129.16, 130.03, 130.21, 130.50, 131.72, 140.98, 154.05 ppm.

Compound 8h

Solid; M.P.: 174–176 °C; IR (KBr): 3494 (–OH stretching), 1750 (C=O Stretching), 1622 (C=C Stretching), 1490, 1398, 1164, 1066 (C–O Stretching) cm^{-1}; ^{1}H-NMR: (300 MHz, DMSO- d_6): δ 2.16 (s, 6H), 4.57–4.64 (m, 4H), 4.74 (s, 1H), 6.88 (d, 2H, $J=3.2$ Hz), 6.97 (d, 1H, $J=7.6$ Hz), 7.94 (brs, 2H); ^{13}C NMR (75 MHz, DMSO-d_6): δ 19.12, 20.38, 33.02, 68.14, 98.97, 125.26, 128.40, 129.89, 134.08, 136.60, 138.61, 176.08, 176.20 ppm.

Compound 9a

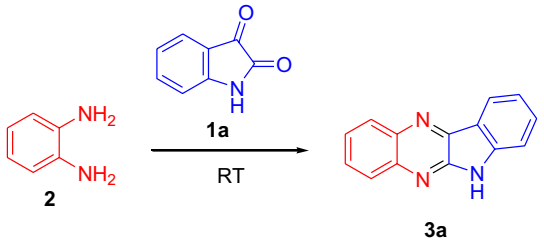
Solid; M.P.: 169–171 °C; IR (KBr): 3408 (-OH stretching), 3160 (N–H stretching), 3078, 1744 (lactone C=O Stretching), 1561 (lactum C=O Stretching), 1326, 924, 731 cm^{-1}; ^{1}H-NMR (300 MHz, DMSO-d_6): δ 4.60 (s, 4H), 6.74 (d, 1H, $J=7.8$ Hz), 6.86 (t, 1H, $J=7.6$ Hz), 7.08 (t, 1H, $J=7.5$ Hz), 7.46 (d, 1H, $J=7.5$ Hz), 10.68 (s, 1H), 10.82 (brs, 2H); ^{13}C NMR (75 MHz, DMSO-d_6): δ 48.24, 67.02, 109.21, 122.02, 127.12, 128.60, 131.06, 142.05, 172.54, 174.85, 178.12 ppm.

Results and discussion

In order to optimize the reaction conditions, we carried out the condensation of isatins and o-phenylene diamine at ambient temperature (Table 1). Previously, very few catalytic systems, such as tetrabutylammonium bromide (TBAB) [43], glacial acetic acid [44] and sulfamic acid [45] have been reported to be effective for the synthesis of indoloquinoxaline derivatives. However, these methods have much or less shortcomings which limit their use under the aspect of environmentally benign processes.

To show the efficacy of Brönsted acid hydrotrope combined catalyst (BAHC), we initially studied the synthesis of indoloquinoxaline by reacting isatin (**1a**) and o-phenylene diamine (**2**). At room temperature in the absence of catalyst in water, above reaction did not furnish the desired indoloquinoxaline **3a** (Table 1, entry 1). When the model reaction was carried out in the presence of *p*-Toluene Sulfonic Acid (PTSA) (10 mol %), the reaction gave **3a** with 40% yield in 6.0 h (Table 1, entry

Table 1 Screening of various reaction conditions for the synthesis of indoloquinoxaline[a]



Entry	Hydrotrope	Aqueous hydrotrope concentration (% *w/v*)	PTSA (mol %)	Time (h)	Yield[b] (%)
1	H_2O	–	–	6.0	00
2	H_2O	–	10	6.0	40
3	NaPTS	20	–	3.0	55
4	NaPTS	20	10	2.5	71
5	NaPTS	10	10	2.5	61
6	NaPTS	30	10	2.0	82
7	NaPTS	40	10	100 min	89
8	NaPTS	50	10	100 min	89
9	NaXS	40	10	100 min	87
10	NaBS	40	10	100 min	86
11	NaCuS	40	10	110 min	85
12	NaSaI	40	10	110 min	86

[a]Reaction conditions: Isatin (1 mmol), o-phenylene diamine (1 mmol), PTSA (10 mol%), aqueous hydrotropic solution (5 mL), RT

[b]Isolated yields

2). However, the addition of NaPTS (sodium p-toluene sulfonate) hydrotrope in the presence of 10 mol % PTSA afforded a 71% product yield within 2.5 h in 20% *w/v* hydrotropic concentration (Table 1, entry 4). On the completion of the reaction monitored by TLC, the reaction mixture was diluted with cold water and product was separated out. From this optimization study, we concluded that hydrotrope aided acid catalysis showed significant progress in product yield at room temperature.

Next, the model reaction was performed with various concentrations of NaPTS including 10–50% *w/v* in water (Table 1, entries 5–8). By changing the hydrotropic concentration, a dramatic effect on the yield of product was observed. As shown in Table 1, product yield changes linearly with concentration. The yield of the product was highest at 40% of hydrotropic concentration, since this concentration may be optimum for the maximum solubilization of organic reactants (Table 1, entry 7). On further increasing hydrotropic concentration up to 50% *w/v*, the isolated product remains as usual as 89% yield. (Table 1, entry 8). Subsequently, in order to examine the effectiveness of the NaPTS, we used various hydrotropes such as NaXS (sodium

xylene sulfonate), NaBS (sodium benzene sulfonate), NaCuS (sodium cumene sulphonate) and NaSaI (sodium salicylate) with 10 mol % PTSA. The reaction results revealed that compared to NaXS, NaBS, NaCuS and NaSaI, the reaction proceeds with high efficiency when PTSA was combined with NaPTS (Table 1, entries 9–12). Thus, it is worth noting that the reaction gave 89% yield of product within 100 min reaction time. The higher activity of NaPTS observed due to its overall planar structure and presence of more balanced hydrophobic and hydrophilic regions that offer a good micro-environment of lower polarity and stabilizes the reactants through a cooperative binding mechanism [42].

With the optimized reaction condition in hand, we expanded the scope and generality of reaction by reacting o-phenylene diamine (**2**) with various substituted isatins (**1a-f**) containing electron-withdrawing or electron-donating groups (Table 2). It was observed that the isatin with electron-donating groups reacted faster and gave higher yields (Table 2, entries 2, 3 and 5) as compared to isatin with electron-withdrawing groups (Table 2, entries 4 and 6).

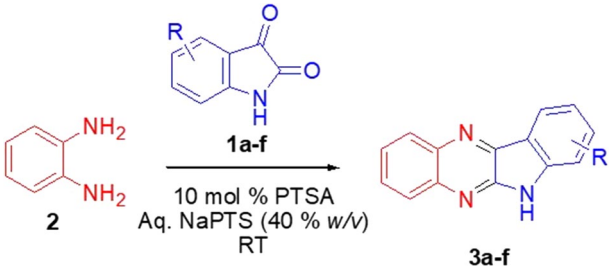
Further, under the similar reaction conditions, ninhydrine (**4a**) and acenaphthoquinone (**4b**) also underwent smooth condensation with o-phenylene diamine (**2**) to furnish the corresponding quinoxaline derivatives (**5a and 5b**) in good yields (Scheme 2).

Encouraged by the above-mentioned results, we decided to carry out the synthesis of bis-tetronic acids using BAHC in water under similar reaction condition. The detailed literature survey showed that there are only few reports available for the synthesis of bis-tetronic acids. Zhang et al. [46] described the preparation of bis-tetronic acids using aldehydes as starting compounds and diethylamine as well as an electrochemically generated base (EGB) as the catalysts. Daribi and coworkers [47] reported the synthesis of bis-tetronic acids using isatin as starting compounds, while Desai et al. [48] synthesized bis-tetronic acids by condensation reaction of aldehydes/isatins and tetronic acid using sulfamic acid as a solid acid catalyst. In our initial endeavor to synthesize bis-tetronic acids (**8a**), we carried out the condensation of benzaldehyde (**6**) (1 mmol) with tetronic acid (**7**) (2 mmol) in a freshly prepared BAHC (10 mol % PTSA and 5 mL 40% *w/v* NaPTS) in water at room temperature. The complete transformation of the desired product **8a** was observed in 3.5 h, as monitored by TLC with isolated 88% yield (Table 3, entry 1). We also investigated the substrate scope by reacting different aldehydes and isatins with tetronic acid. It was observed that all the aromatic aldehydes and isatins furnished corresponding products (**8a-h** and **9a-f**) in excellent yields (Table 3, entries 1–14).

Reaction mechanism for synthesis of bis-tetronic acids

A mechanistic rationale portraying the plausible progress of events is briefly outlined in Scheme 3. PTSA probably activates the carbonyl carbon of aldehyde (**6**), which undergoes the nucleophilic attack of tetronic acid **(7)** via Knoevenagel condensation to give α,β-unsaturated dicarbonyl intermediate (**A**). Subsequently, another tetronic acid (**7**) molecule attack intermediate (**A**), giving Michael adducts (**B**) which upon tautomerization yields the desired product (**8**).

Table 2 Synthesis of quinoxalines in BAHC[a]



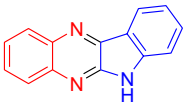
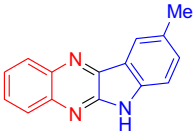
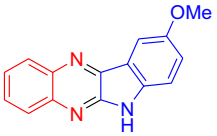
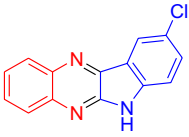
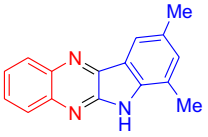
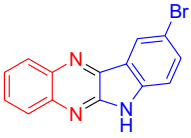
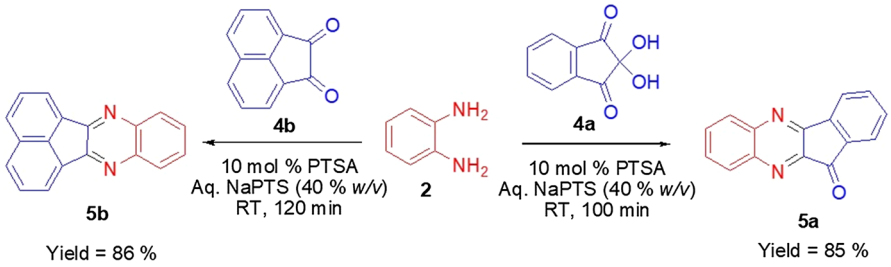
Entry	Product	Time (min)	Yield[b] (%)	Melting point (°C)	
				Obs	**Lit.**[Ref.]
1	 **3a**	100	89	285–287	290–292 [45]
2	 **3b**	90	89	291–292	> 293 [45]
3	 **3c**	90	90	302–304	> 300 [45]
4	 **3d**	120	84	220–222	222–224 [45]
5	 **3e**	90	89	301–303	> 300 [45]
6	 **3f**	120	83	> 300	> 300 [45]

Table 2 (continued)

[a]Reaction conditions: Isatins (1 mmol), o-phenylenediamine (1 mmol), PTSA (10 mol %), 40% aqueous NaPTS hydrotropic solution (5 mL), RT



Scheme 2 Synthesis of quinoxaline of ninhydrine / acenaphthoquinone in BAHC

In this reaction, the eliminated water molecules get easily absorbed by hydrophilic head groups of hydrotrope. As a result of the overall effect, there is rate enhancement of reaction.

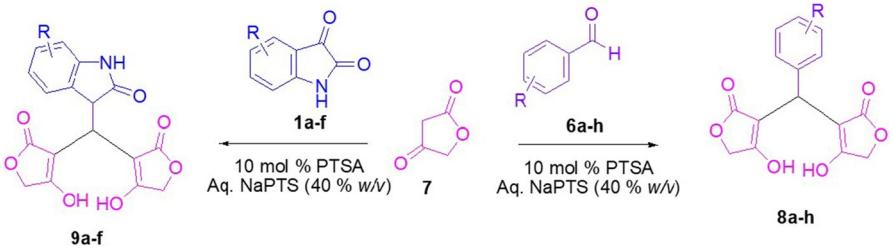
Recyclability study of BAHC

Hydrotropes being a novel class of reaction media which is widely used to develop a highly efficient protocol for the preparation of various heterocyclic compounds. In addition, BAHC could be easily reused many times. Thus, the recyclability of the BAHC was studied for two reactions. After the completion of the reaction, water was added to the reaction mixture and the precipitated product was simply separated by filtration. The aqueous solution of BAHC was recovered by filtration, washed thoroughly with diethyl ether, concentrated and then used for the next reaction cycle. As shown in Fig. 2, the BAHC could be recycled up to five times without a significant decrease in catalytic properties for both the reactions.

Conclusion

In summary, we have synthesized indoloquinoxaline and bis-tetronic acid derivatives by a simple and sustainable one-pot approach using Brönsted acid hydrotrope combined catalyst (BAHC) in water at room temperature. The developed method provides several advantages like clean reaction profiles, water as a reaction medium, reactions at ambient temperature, easy isolation of product and high yield of products. Moreover, the BAHC could be recycled at least five times.

Table 3 Synthesis of bis-tetronic acids in BAHC[a]



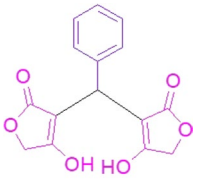
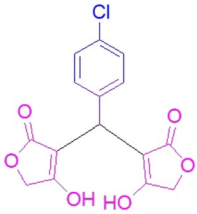
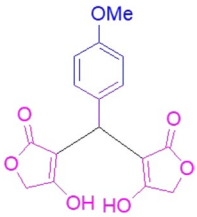
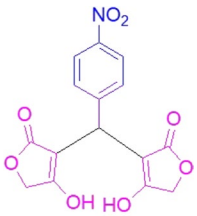
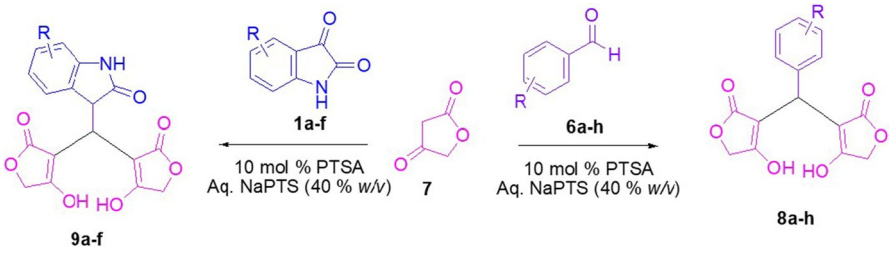
Entry	Product	Time (h)	Yield[b] (%)	Melting point (°C)	
				Obs	**Lit.**[Ref.]
1	 **8a**	3.5	88	131–133	132–135 [48]
2	 **8b**	3.0	84	213–215	210–212 [48]
3	 **8c**	3.0	89	170–172	174–178 [48]
4	 **8d**	3.0	83	232–234	232–234 [48]

Table 3 (continued)



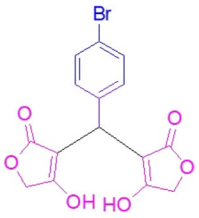
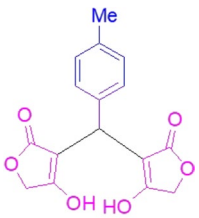
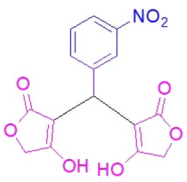
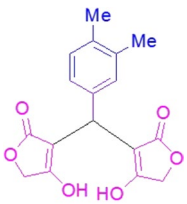
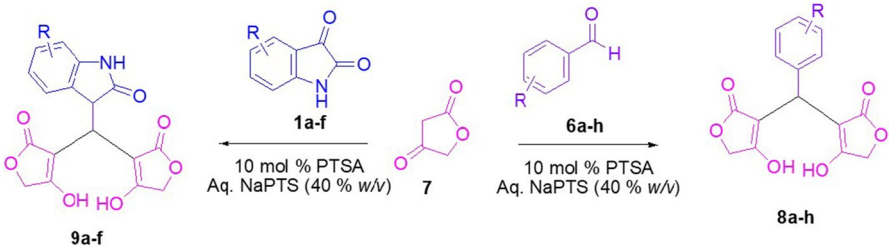
Entry	Product	Time (h)	Yield[b] (%)	Melting point (°C)	
				Obs	**Lit.**[Ref.]
5	 **8e**	3.0	83	190–192	187–190 [48]
6	 **8f**	3.5	84	191–193	190–192 [48]
7	 **8g**	2.5	86	240–242	235–237 [48]
8	 **8h**	3.0	87	174–176	175–178 [48]

Table 3 (continued)



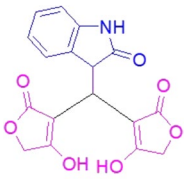
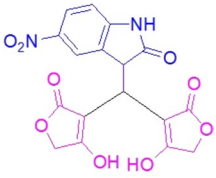
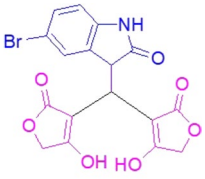
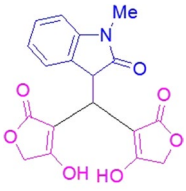
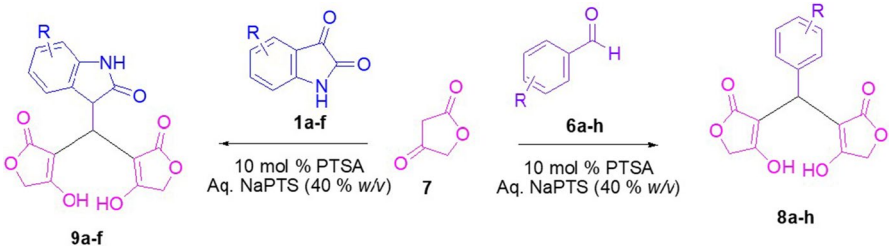
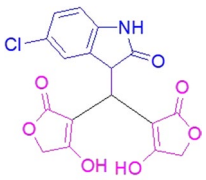
Entry	Product	Time (h)	Yield[b] (%)	Melting point (°C)	
				Obs	**Lit.**[Ref.]
9	 **9a**	4.0	88	169–171	168–170 [48]
10	 **9b**	3.5	85	178–180	175–178 [48]
11	 **9c**	3.5	84	180–182	182–184 [48]
12	 **9d**	4.0	85	160–162	158–161 [48]

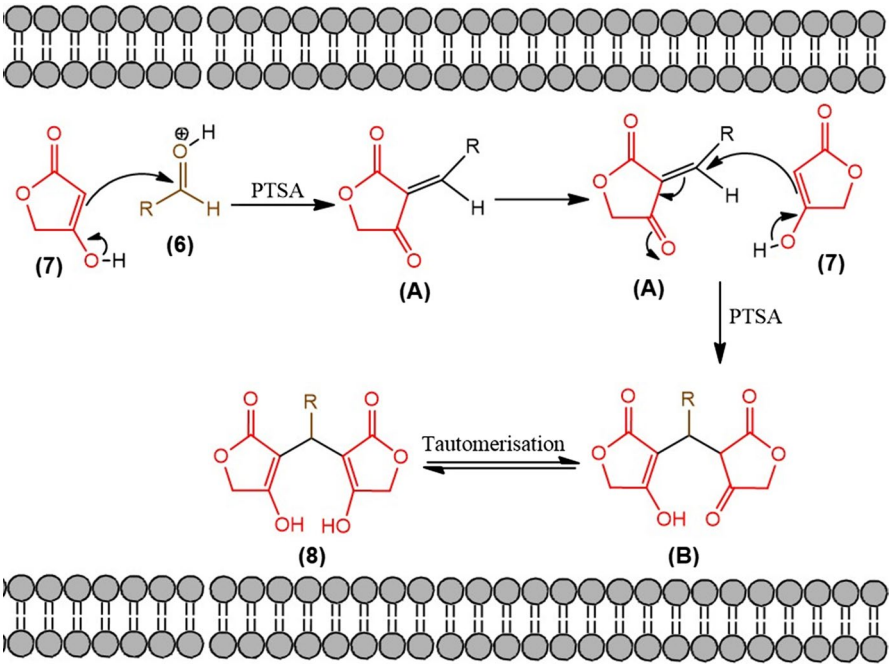
Table 3 (continued)



Entry	Product	Time (h)	Yield[b] (%)	Melting point (°C)	
				Obs	**Lit.**[Ref.]
13	 **9e**	4.0	85	162–164	162–164 [48]
14	 **9f**	4.0	86	170–172	177–179 [48]

[a]Reaction conditions: Aldehydes/isatins (1 mmol), tetronic acid (2 mmol), PTSA (10 mol%), 40% aqueous NaPTS hydrotropic solution (5 mL), RT

[b]Isolated yields



Scheme 3 Plausible mechanistic pathway for synthesis of bis-tetronic acids in BAHC

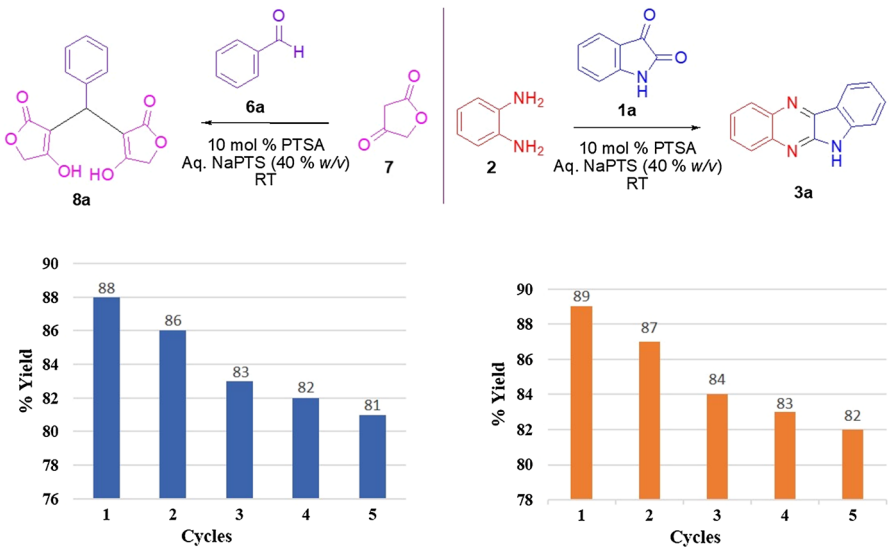


Fig. 2 Recyclability study of BAHC catalyst

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