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ORIGINAL ARTICLE

Application of sulfonic acid functionalized nanoporous silica (SBA-Pr-SO_3H) in the green one-pot synthesis of triazoloquinazolinones and benzimidazoquinazolinones

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KEYWORDS

Sulfonic acid functionalized SBA-15;
Triazoloquinazolinones;
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Nanoporous solid acid catalyst;
Solvent free condition

Abstract Sulfonic acid functionalized SBA-15 (SBA-Pr-SO_3H) with a pore size of 6 nm was proven to be an efficient heterogeneous nanoporous solid acid catalyst in the green one-pot synthesis of triazoloquinazolinones and benzimidazoquinazolinones from the reaction of aromatic aldehydes with 3-amino-1,2,4-triazole (or 2-aminobenzimidazole) and dimedone under solvent free conditions.

1. Introduction

In recent times, solid acid catalysts such as ordered mesoporous materials (Díaz et al., 2000; Maheswari et al., 2003) have received a considerable attention in organic synthesis because of their environmental compatibility, reusability, high selectivity, operational simplicity, non-corrosiveness and ease of isolation of the products. In 1998, high surface area, large pore size, greater pore wall thickness, hydrothermally robust and hexagonally ordered mesoporous SBA-15 silica was synthesized (Zhao et al., 1998a,b). Since then, SBA-15 was modified to suit different catalytic applications (Burri et al., 2002). It can be used as catalyst (Mohammadi Ziarani et al., 2007; Trong et al., 2001) for the preconcentration of metals (Ganjali et al., 2006a,b, 2004), and as modified carbon electrodes (Badiei et al., 2005; Walcarius et al., 1999; Zhang et al., 2006). Accordingly, SBA-15 was modified as a solid acid catalyst with sulfonic acid functionalization (SBA-Pr-SO_3H). Integration of acidic functional groups (e.g., –SO_3H) into SBA-15 has been explored to produce promising solid acids (Margolese et al., 2000). Applications of these nanocatalysts in organic synthesis and one-pot reaction in green conditions are so important.

Quinazoline 1, medicinally, has been used in various areas especially as an anti-malarial agent and in cancer treatment. One example of a compound containing the quinazoline

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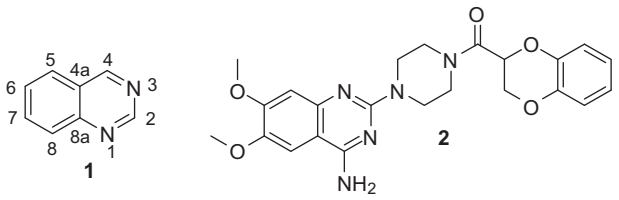
structure is doxazosin mesylate 2 which is an alpha blocker used to treat high blood pressure and benign prostatic hyperplasia (BHP).



Quinazolinone derivatives have many biological activities such as analgesic, anti-inflammatory, antipyretic (Agarwal et al., 1988; Bekhit and Khalil, 1998; Daidone et al., 1994), antimicrobial (Prameela et al., 1992), anticonvulsant (Shrimali et al., 1991), fungicidal (Shakhidoyator et al., 1980), antidepressant (Fetter et al., 1991) and antitumor compounds (Braña et al., 1997). In the literature, a few methods for the synthesis of these compounds have been reported using different conditions such as MW radiation (Mourad et al., 2007; Shao et al., 2008), several solvents (Insuasty et al., 2004; Lipson et al., 2003a,b) and catalysts including $H_6P_2W_{18}O_{62}$ (Heravi et al., 2008), NH_2SO_3H (Heravi et al., 2010), and ionic liquid (Yao et al., 2010). But at this time, it is important to develop a more efficient and greener method in the synthesis of triazoloquinazolinones and benzimidazoquinazolinones using nano-acid catalysts. There are only a few reports about the application of several types of sulfonic acid functionalized ordered mesoporous silicas as nanoacid catalyst in chemical transformations (Das et al., 2006; Karimi and Zareyee, 2008; Kureshy et al., 2009; Sreevardhan Reddy et al., 2007; Van Rhijn et al., 1998). In continuation of our studies, on the application of heterogeneous solid catalysts to organic synthesis (Mohammadi Ziarani et al., 2011, 2008, 2010), in this paper we want to report the application of SBA-Pr-SO_3H as a highly active nanoporous heterogeneous acid catalyst in the preparation of triazoloquinazolinones and benzimidazoquinazolinones.

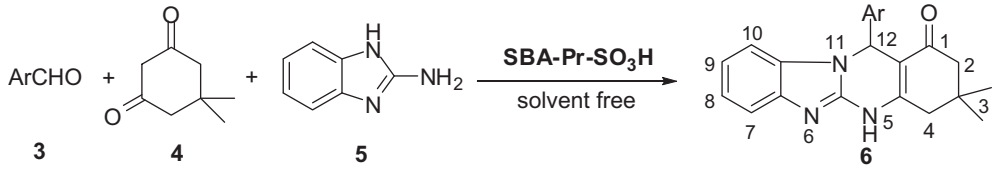
2. Results and discussion

In this paper, the condensation of aromatic aldehydes with 3-amino-1,2,4-triazole (or 2-aminobenzimidazole) and dimedone in the presence of nanoporous solid acid catalyst (SBA-Pr-SO_3H) for the preparation of quinazolinones derivatives has been studied (Schemes 1 and 2). We initially investigated the solvent effects in this reaction. As shown results in Table 1, among the tested solvents such as H_2O, DMF, CH_3CN, and solvent-free system, the best result was obtained after 5–10 min in solvent-free condition in excellent yield. Therefore, this reaction was developed with different aldehydes and the results were summarized in the Table 2. The time of reaction was within 5–15 min and high yields of triazoloquinazolinones and benzimidazoquinazolinones were obtained. After completion of the reaction (monitored by TLC), the crude product was dissolved in hot ethanol, the heterogeneous solid catalyst was removed easily by simple filtration, and after cooling of the filtrate, the pure crystals of products were obtained. The acid catalyst can be reactivated by simple washing subsequently with diluted acid solution, water and acetone, and then reused without noticeable loss of reactivity. The new products were characterized by IR and NMR spectroscopy data for new compounds. Melting points are compared with reported values in the literature as shown in Table 2.

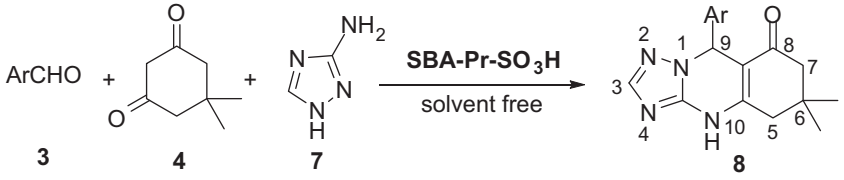
The suggested mechanism for the SBA-Pr-SO_3H catalyzed transformation is shown in Schemes 3 and 4. Concerning the reaction mechanism, we suggest that initially, the solid acid catalyst protonates the carbonyl group of aldehyde, which then condenses with dimedone to produce the adduct product

Table 1 The optimization of reaction conditions in the synthesis of triazoloquinazolinones/benzimidazoquinazolinones.

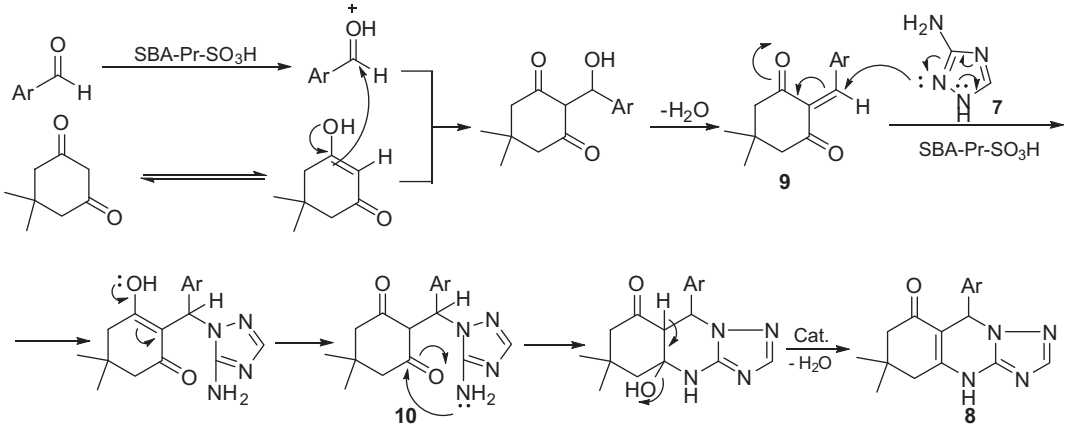
No.	Amine	Solvent	Time (min)	Yield (%)
1	3-Amino-1,2,4-triazole	CH_3CN	35	78
2	3-Amino-1,2,4-triazole	DMF	40	70
3	3-Amino-1,2,4-triazole	H_2O	50	80
4	3-Amino-1,2,4-triazole	–	5	90
5	2-Aminobenzimidazole	CH_3CN	30	78
6	2-Aminobenzimidazole	DMF	40	75
7	2-Aminobenzimidazole	H_2O	55	85
8	2-Aminobenzimidazole	–	10	90



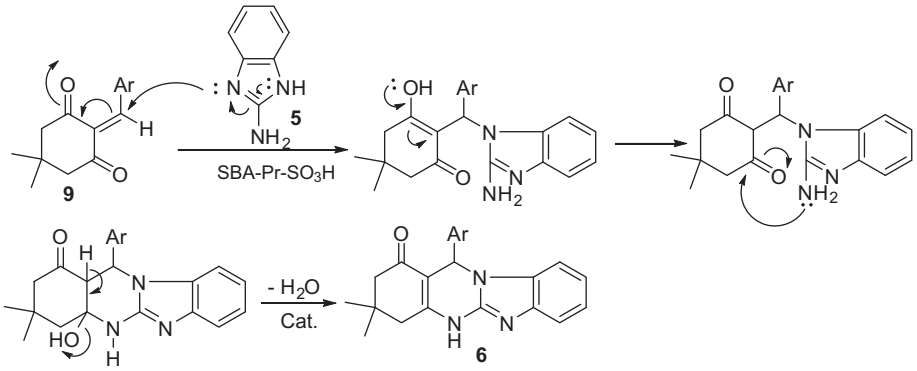
Scheme 1 Synthesis of 12-(aryl)-3,3-dimethyl-2,4,5,12-tetrahydrobenzimidazo [1,2-*b*]quinazolin-1-one in the presence of SBA-Pr-SO_3H.



Scheme 2 Synthesis of 9-(aryl)-6,6-dimethyl-5,6,7,9-tetrahydro-4H-[1,2,4]-triazolo-[5,1-b]-quinazolin-8(4H)-ones in the presence of SBA-Pr-SO_3H.



Scheme 3 Proposed mechanism for the synthesis of [1,2,4]-triazolo-quinazolinone derivatives.



Scheme 4 Proposed mechanism for the synthesis of benzimidazoquinazolinones derivatives.

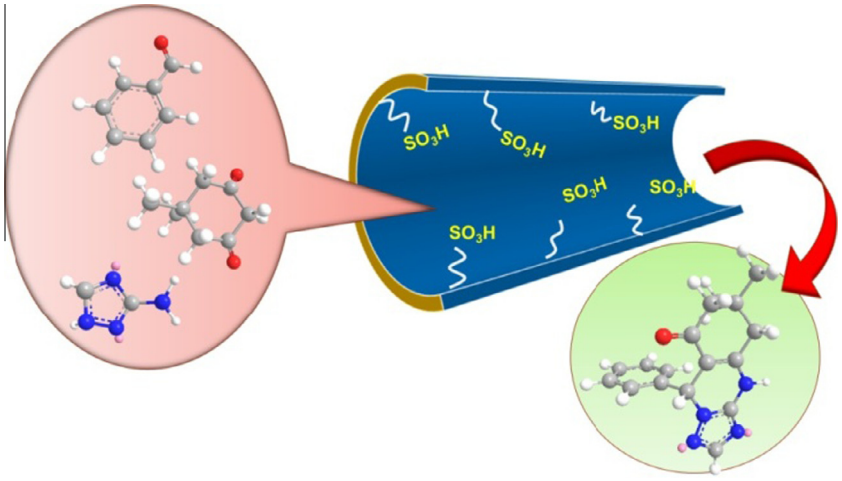


Figure 1 SBA-Pr-SO_3H acts as a nano-reactor.

9. Michael addition reaction of nitrogen number 3 in 2-amino-benzimidazole **5** or nitrogen number 2 in 3-amino-1, 2, 4-triazole **7** to adduct product **9** creates intermediate **10** and **11**. The reaction of amino group with carbonyl group of **10** or **11** and then elimination of water produce the desired enamine products **6**, **8** in excellent yield. The high yields of reactions are attributed to the effect of nanopore size about 6 nm of solid acid catalyst, which could act as nano-reactor (Fig. 1).

The syntheses of triazoloquinazolinones and benzimidazoquinazolinones have been studied with several catalysts and solvents in the literature as shown in Tables 3 and 4. In contrast with other existing methods, the present methodology

offers several advantages such as excellent yields, a simple procedure, short reaction times, easy synthesis, simple work-up and greener conditions.

2.1. Preparation of catalyst

The SBA-15 as a new nanoporous silica can be prepared by using commercially available triblock copolymer Pluronic P126 as a structure directing agent (Zhao et al., 1998a,b). Integration of acidic functional groups (e.g., $–SO_3H$) into SBA-15 has been explored to produce promising solid acids. The sulfonic acid functionalized SBA-15 was usually synthesized through direct synthesis or post-grafting (Lim et al., 1998; Wight and Davis, 2002). A schematic illustration for the preparation of SBA-Pr-SO_3H was shown in Fig. 2. First, the calcined SBA-15 silica was functionalized with (3-mercaptopropyl)trimethoxysilane (MPTS) and then, the thiol groups were oxidized to sulfonic acid by hydrogen peroxide. The surface of the catalyst was analyzed by different methods such as TGA, BET and CHN methods which demonstrated that the organic groups (propyl sulfonic acid) were immobilized into the pores. The surface area, average pore diameter calculated by the BET method and pore volume of SBA-Pr-SO_3H are 440 $m^2 g^{-1}$, 6.0 nm and 0.660 $cm^3 g^{-1}$, respectively (Table 5), which are smaller than those of SBA-15 due to the immobilization of sulfonosilane groups into the pores (Mohammadi Ziarani et al., 2010).

3. Experimental section: general information

IR spectra were recorded from KBr disk using a FT-IR Bruker Tensor 27 instrument. Melting points were measured by using the capillary tube method with an electro thermal 9200 apparatus. The ^1H NMR (250 MHz) was run on a Bruker DPX, 250 MHz. Nitrogen adsorption and desorption isotherms were measured at −196 °C using a Japan Belsorb II system after the samples were vacuum dried at 150 °C overnight.

3.1. Preparation of SBA-15

The synthesis of SBA-15 was carried out in accordance to the earlier reports (Zhao et al., 1998a,b). In a typical synthesis batch, triblock copolymer surfactant as a template (P123 = $EO_{20}PO_{70}EO_{20}$, M_{ac} = 5800) (4.0 g) was dissolved in 30 g of water and 120 g of 2 M HCl solution. Then, TEOS (tetraethylorthosilicate) (8.50 g) was added to reaction mixture which was stirred for 8 h at 40 °C. The resulting mixture was transferred into a Teflon-lined stainless steel autoclave and kept at 100 °C for 20 h without stirring. The gel composition P123:HCl:H_2O:TEOS was 0.0168:5.854:162.681:1 in molar ratio. After cooling down to room temperature, the product was filtered, washed with distilled water and dried overnight at 60 °C in air. The as-synthesized sample was calcinated at 550 °C for 6 h in air atmosphere to remove the template.

3.1.1. Functionalization of the SBA-15 by organic groups

Functionalization of the SBA-15 catalyst was schematically shown in Fig. 2. The calcinated SBA-15 (2 g) and (3-mercaptopropyl)trimethoxysilane (10 ml) in dry toluene (20 ml) were refluxed for 24 h. The product was filtered and extracted for 6 h in CH_2Cl_2 using a soxhlet apparatus, then dried under vacuum. The solid product was oxidized with H_2O_2 (excess) and one drop of H_2SO_4 in methanol (20 ml) for 24 h at rt and then the mixture was filtered and washed with H_2O, and acetone. The modified SBA-15-Pr-SO_3H was dried and used as nanoporous solid acid catalyst in the following reactions.

3.2. General procedure for the preparation of quinazolinone derivatives

The SBA-Pr-SO_3H (0.05 g) was activated in vacuum at 100 °C and then after cooling to room temperature, 5,5-dimethylcyclohexane-1,3-dione **4** (0.14 g, 1 mmol), aldehyde **3** (1 mmol), and 2-aminobenzimidazole **5** (0.133 g, 1 mmol) or 3-amino-1,2,4-triazole **7** (84 mg, 1 mmol) were added to it. The mixture was heated in solvent free condition for an appropriate time. The completion of reaction was indicated by TLC, the resulting solid product was dissolved in hot ethanol, filtered for removing the unsolvable catalyst and then the filtrate was cooled to afford the pure product. The spectroscopic and analytical data for selected compounds are presented in the following part. The catalyst was washed subsequently with diluted acid solution, distilled water and then acetone, dried under vacuum and re-used for several times without loss of significant activity.

3.2.1. 9-Phenyl-6,6-dimethyl-5,6,7,9-tetrahydro-4H-1,2,4-triazolo[5,1-b]quinazolin-8-one (**8a**)

IR (KBr): υ_{max} = 3416, 3224, 3031, 2961, 2926, 2837, 1648, 1582, 1547, 1473, 1453, 1414, 1368, 1334, 1254, 729, 697 cm^{-1}. ^1H NMR (250 MHz, DMSO-d_6) δ_H = 0.96 (s, 3H, CH_3), 1.04 (s, 3H, CH_3), 2.04–2.26 (q, AB, 2H, H-5), 2.49–2.55 (m, 2H, H-7), 6.20 (s, 1H, H-9), 7.17–7.31 (m, 5H, Ar-CH), 7.68 (s, 1H, H-3), 11.13 (s, 1H, NH) ppm.

3.2.2. 6,6-Dimethyl-9-(4-methoxyphenyl)-5,6,7,9-terahydro-4H-1,2,4-triazolo[5,1-b]quinazolin-8-one (**8d**)

IR (KBr): υ_{max} = 3446, 3150–2760 (br), 3093, 2961, 2930, 1735, 1646, 1585, 1511, 1463, 1416, 1367, 1247, 1145, 1031, 803 cm^{-1}. ^1H NMR (250 MHz, DMSO-d_6) δ_H = 0.94–0.97 (d, 6H, $2CH_3$), 2.19–2.26 (d.d, 2H, H-5), 2.32 (br, 2H, H-7), 3.67 (s, 3H, OCH_3), 6.15 (s, 1H, H-9), 6.72–6.88 (m, 4H, Ar-CH), 7.67 (s, 1H, H-3), 11.08 (s, 1H, NH) ppm.

3.2.3. 12-(4-Chlorophenyl)-3,3-dimethyl-2,4,5,12-tetrahydrobenzimidazo[1,2-b]quinazolin-1-one (**6b**)

IR (KBr): υ_{max} = 3442, 3119, 3081, 3030, 2959, 2925, 2890, 1646, 1581, 1550, 1528, 1482, 1416, 1350, 1254, 730, 695 cm^{-1}. ^1H NMR (250 MHz, DMSO-d_6) δ_H = 0.88 (s, 3H, CH_3), 1.01 (s, 3H, CH_3), 2.12–2.25 (m, 2H), 2.45–2.56 (m, 1H, H-2), 2.63–2.68 (m, 1H, H-2), 6.39 (s, 1H, H-12), 6.89–7.04 (m, 4H, Ar-CH), 7.13–7.18 (m, 2H, ArCH), 7.21–7.35 (m, 2H, Ar-CH), 11.13 (s, 1H, NH) ppm.

4. Conclusion

In summary, a novel and highly efficient method for the synthesis of triazoloquinazolinones and benzimidazoquinazolinones has been achieved by the one-pot, three component reaction of aromatic aldehydes with 3-amino-1,2,4-triazole (or 2-aminobenzimidazole) and dimedone under solvent free conditions using the reusable and environmentally benign sulfonic acid functionalized nanoporous silica (SBA-Pr-SO_3H) as

Table 2 SBA-Pr-SO_3H catalyzed the synthesis of triazoloquinazolinones **8**/benzimidazoquinazolinones **6** under solvent-free condition.

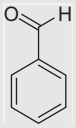
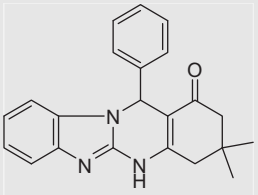
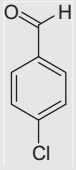
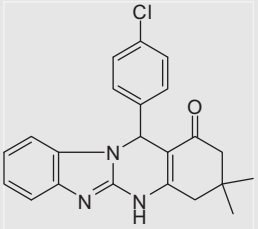
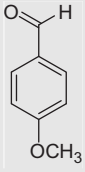
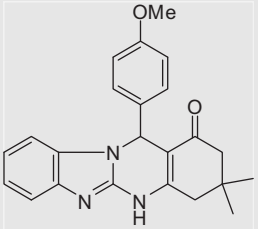
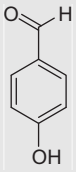
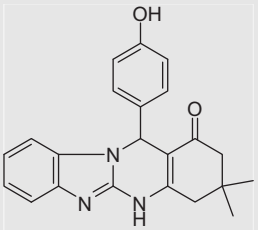
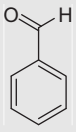
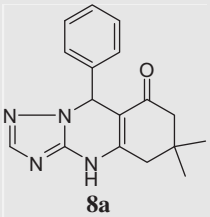
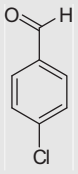
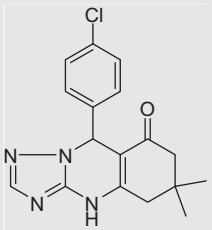
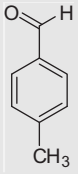
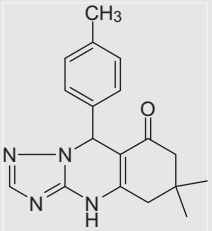
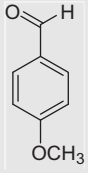
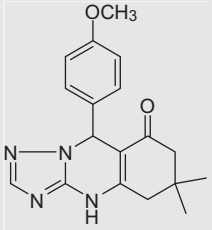
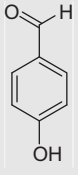
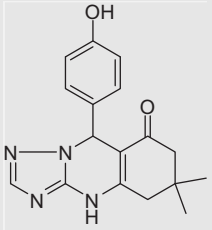
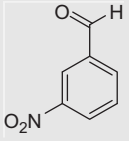
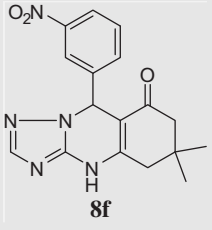
Entry	Aldehyde	Product	Time (min)	Yield%	mp (°C)	mp (L)
1		 **6a**	10	90	320–322	322–324 (Mourad et al., 2007)
2		 **6b**	10	93	337–340	340 (Mourad et al., 2007)
3		 **6c**	15	87	317–319	318–320 (Mourad et al., 2007
4		 **6d**	15	87	330–332	> 300 (Heravi et al., 2008)
5		 **8a**	5	90	247–250	248–250 (Lipson et al., 2003a,b)

Table 2 (*continued*)

Entry	Aldehyde	Product	Time (min)	Yield%	mp (°C)	mp (L)
6		 **8b**	5	90	280–282	281–283 (Lipson et al., 2003a,b)
7		 **8c**	8	87	281–282	280–282 (Mourad et al., 2007)
8		 **8d**	8	88	223–224	222–224 (Lipson et al., 2003a,b)
9		 **8e**	10	85	318–320	> 300 (Lipson et al., 2003a,b)
10		 **8f**	5	96	265–267	266–269 (Lipson et al., 2003a,b)

a nano and green solid acid catalyst under solvent-free conditions. The attractive features of this protocol are simple procedure, short reaction time, high yields, simple workup, the reusability of catalyst and non-chromatographic purification of products, i.e., simple recrystallization from ethanol. The best yield and short reaction time are related to the high

Table 3 Comparison of different conditions in the synthesis of triazoloquinazolinones **8**.

Entry	Catalyst	Solvent	Condition	Time (min)	Yield	Year	Ref.
1	–	DMF	Reflux	30	48–76	2003	(Lipson et al., 2003a,b)
2	–	DMF	Reflux	30–90	62–76	2007	(Mourad et al., 2007)
3	–	DMF	M.W.	3–10	92–95	2007	(Mourad et al., 2007)
4	$H_6P_2W_{18}O_{62}$	CH_3CN	Reflux	30–60	90–97	2008	(Heravi et al., 2008)
5	NH_2SO_3H	CH_3CN	Reflux	25–60	89–96	2010	(Heravi et al., 2010)
6	SBA-Pr-SO_3H	–	Heating	5–10	85–96	This work	

Table 4 Comparison of different conditions in the synthesis of benzimidazoquinazolinones **6**.

Entry	Catalyst	Solvent	Condition	Time (min)	Yield	Year	Ref.
1	–	DMF	Reflux	5	53–65	2003	(Lipson et al., 2003a,b)
2	–	DMF	Reflux	6–12 h	64–72	2007	(Mourad et al., 2007)
3	–	DMF	M.W.	1–5	85–96	2007	(Mourad et al., 2007)
4	$H_6P_2W_{18}O_{62}$	CH_3CN	Reflux	10–20	91–99	2008	(Heravi et al., 2008)
5	NH_2SO_3H	CH_3CN	Reflux	15–20	90–95	2010	(Heravi et al., 2010)
6	–	H_2O	M.W.	2–4	91–97	2008	(Shao et al., 2008)
7	Ionic liquid	–	Heating	6–7 h	82–86	2010	(Yao et al., 2010)
8	–	EtOH	Reflux	15–90	64	2004	(Insuasty et al., 2004)
9	SBA-Pr-SO_3H	–	Heating	10–15	87–93		This work

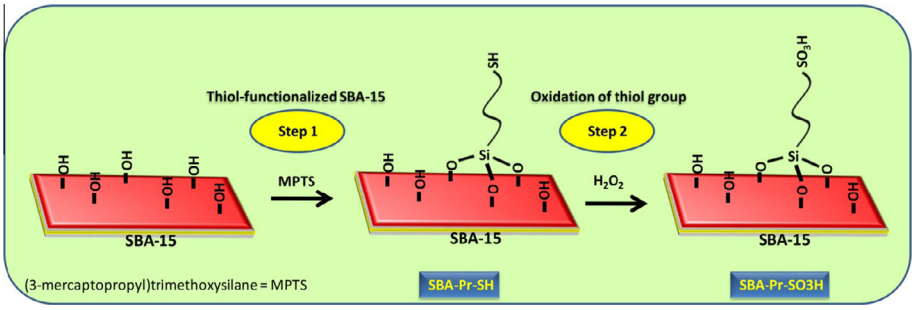


Figure 2 Schematic illustration for the preparation of SBA-Pr-SO_3H.

Table 5 Porosimetery values for SBA-15 and functionalized SBA-15.

	Surface area ($cm^2\ g^{-1}$)	Pore volume ($cm^3\ g^{-1}$)	Pore diameter (nm)
SBA-15	649	0.806	6.2
SBA-SO_3H	440	0.660	6.0

efficiency of the nano-catalyst of SBA-Pr-SO_3H with the pore size of 6 nm. The results demonstrated that the reaction takes place easily in the nano-pores of the catalyst.

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