Res Chem Intermed
DOI 10.1007/s11164-015-2096-0

Efficient *N*-Boc protection of amines by a reusable heterogeneous solid acid nanocatalyst at room temperature

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Received: 9 March 2015 / Accepted: 6 May 2015

Abstract An efficient and rapid protocol for chemoselective *N*-Boc protection of various structurally different aryl, aliphatic, and heterocyclic amines is reported with $(Boc)_2O$ using mesoporous silica phenylsulfonic acid (SBA-15-Ph-SO_3H) as a recyclable and heterogeneous solid acid nanocatalyst under solvent-free condition at ambient temperature. The catalyst can be easily recovered and reused for ten reaction cycles for protection of amines without considerable loss of activity. The advantages of this green method are simplicity, easy workup, chemoselectivity, short reaction time, and excellent yield.

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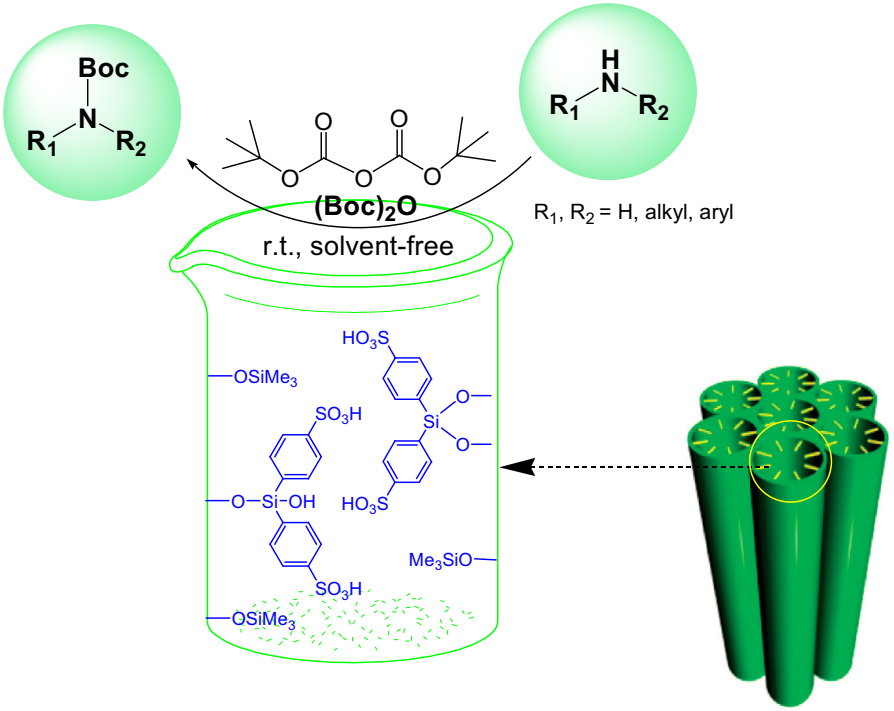
Published online: 12 June 2015

Graphical Abstract



Keywords Nanoreactor · *N*-Boc protection · Mesoporous material · Solid acid catalyst

Introduction

In recent years, organically functionalized ordered mesoporous silicas [1–4] with tunable pore structure, high surface area, and tailored composition have received great attention with broad applications ranging from adsorbents [5–8], gas separation [9], and catalysis [10–15] to biological uses [1, 16]. Some properties of these materials are mechanically stable structure, high surface area, and large, ordered pores with narrow size distribution of an inorganic backbone. This organic mesoporous functionalization has been widely achieved by grafting of functional groups. Ordered mesoporous silicas (OMSs), particularly SBA-15 among other silica materials, have relatively good hydrothermal stability, and possess hexagonal arrays of uniform pores with high special surface area and large pore volume.

Direct synthesis involving co-condensation of siloxane and organosiloxane species in the presence of different templating surfactants has been shown to be a promising alternative to grafting procedures [17–20]. By covalent attachment of sulfonic acid groups to the surface of silica derivatives, several types of solid sulfonic acids, based on ordered mesoporous silicas, have been created in recent

years [21–27]. Silylation and direct synthesis procedures have been used for preparation of these sulfonic-functionalized silica-based materials. The active sulfonic group is obtained postsynthetically by oxidation of propanethiol groups previously anchored to the surface [17, 24–27] or by sulfonation reactions [21, 22].

Protection of amines is a frequently employed strategy for decreasing the nucleophilicity of the amino group in order to perform other transformations in molecules. The presence of an amine function in so many biologically active compounds and organic chemistry makes its protection a frequently needed exercise in synthetic/medicinal chemistry. Among various amine-protecting groups, the *tert*-butoxycarbonyl (Boc) group is perhaps one of the most widely used due to its exceptional stability towards a variety of reagents and reaction conditions yet ready removal under moderately strong acidic conditions [28]. The Boc group has also been used as a directing group for *ortho*-metallation of aromatic amines [29–31]. Di-*tert*-butyl dicarbonate (Boc_2O) [32] is usually the reagent of choice for introduction of Boc groups due to its high reactivity and reasonable stability [33].

Various reagents and methodologies have been developed during recent years for *N*-*tert*-butoxycarbonylation of amines. Most of them are applied in the presence of an organic or inorganic base, for example, $(Boc)_2O$ in the presence of 4-dimethylaminopyridine (DMAP) [34], 4-dimethylamino-1-*tert*-butoxycarbonyl pyridinium chloride/tetrafluoroborate in aq. NaOH [35], 2-*tert*-butyloxycarbonyl oxyimine-2-phenylacetonitrile in the presence of Et_3N in H_2O-dioxane [36], *tert*-butyl-2-pyridyl carbonate in the presence of Et_3N in H_2O-dimethylformamide (DMF) [37] or *tert*-butyl 1-chloroalkyl carbonates in the presence of K_2CO_3 in H_2O-tetrahydrofuran (THF) [38]. However, these methodologies have various drawbacks such as long reaction time, preparation of the *tert*-butoxycarbonylation reagents, and requirement for auxiliary substances (e.g., solvents and other reagents). Furthermore, the base-catalyzed reactions are often associated with formation of isocyanate [39], urea [40], and *N*,*N*-di-Boc derivatives [41]. Moreover, the high toxicity of DMAP and reagents derived from it limits their use [41]. These disadvantages can be avoided by electrophilic activation of $(Boc)_2O$ in the presence of acids. There are examples of other modified methods for *N*-*tert*-butoxycarbonylation of amines, using $H_3PW_{12}O_{40}$ [42], H_2NSO_3H [43], $Zn(ClO_4)_2{\cdot}6H_2O$ [44], $ZrCl_4$ [45], $LiClO_4$ [46], $Cu(BF_4)_2$ [47], Montmorillonite K10 [48], sulfonic-acid-functionalized silica [49, 50], I_2 [51], indium(III) halides [52], tetrafluoroethylene (TFE) [53], protic ionic liquid [TMG][Ac] [54], and ionic liquid 1,3-disulfonic acid imidazolium hydrogen sulfate [55]. The design of new, milder and effective methods for *N*-Boc protection remains an active topic in synthetic chemistry.

Experimental

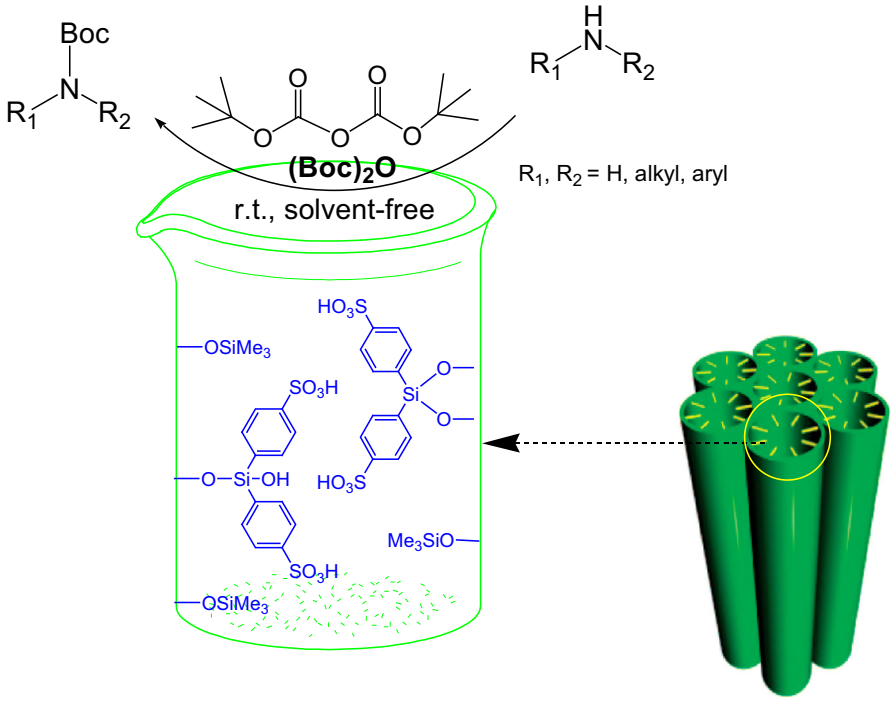
Preparation of SBA-15-Ph-SO_3H

To a 100-mL round-bottomed flask were introduced 30 mL anhydrous toluene and 1.0 g SBA-15, and 5 mL dichlorodiphenylsilane (DCDPS) was added. The solution was refluxed for 12 h under inert atmosphere; the solid was filtered and washed

sequentially with toluene, dichloromethane, and methanol, then dried under reduced pressure at 60 °C for 12 h. Phenyl-modified SBA-15 was dispersed in dry hexane (50 mL) under nitrogen, then trimethylsilylchloride (TMSC, 3 mL) was added to the dispersion, which was cooled to r.t. for 8 h, filtered, and washed with hexane to obtain trimethylsilylated phenyl-modified SBA-15. The dry white solid was soaked in $ClSO_3H$ solution (0.6 mL) in dry $CHCl_3$ (20 mL), and the reaction was refluxed for 2 h. After being filtrated and washed with dry $CHCl_3$, the solid was dried in vacuum at 60 °C for 10 h to obtain SBA-15 functionalized with phenylsulfonic acid groups, designated as SBA-15-Ph-SO_3H. The sulfonic content (number of H^+) of the catalyst based on CHN analysis and titration with NaOH was estimated to be 2.45 mmol/g.

General procedure for the *N*-Boc protection of amines and amino acids in presence of SBA-15-Ph-SO_3H under solvent-free condition

An amine (1 mmol) was added to a magnetically stirred mixture of SBA-15-Ph-SO_3H (1 mol %, 4 mg) and $(Boc)_2O$ (1.1 mmol) at room temperature. The progress of the reaction was monitored by thin-layer chromatography (TLC). After completion of the reaction, the reaction mixture was diluted with EtOH (5 mL) and centrifuged. Then the clear liquid was separated, and the residue containing the



Scheme 1 Protection of amines with di-Boc catalyzed by SBA-15-Ph-SO_3H

Table 1 Protection of amines with di-Boc catalyzed by SBA-15-Ph-SO_3H at room temperature

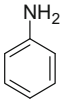
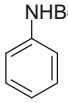
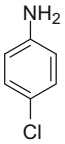
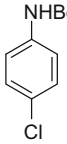
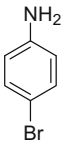
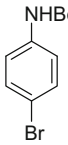
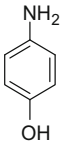
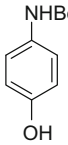
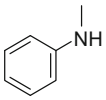
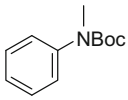
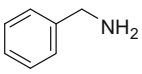
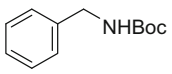
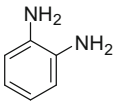
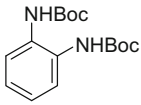
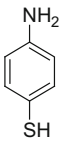
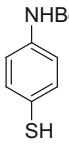
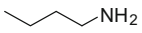
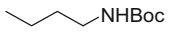
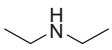
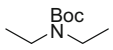
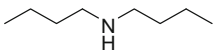
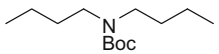
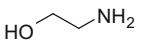
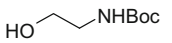
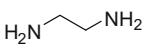
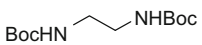
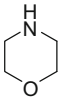
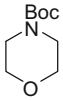
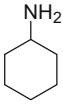
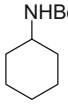
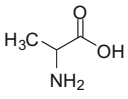
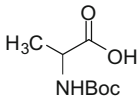
Entry	Substrate	Product	Time (min)	Yield (%)[a]
1			2	98 [69]
2			15	92 [51]
3			15	95 [51]
4			5	98 [70]
5			5	98 [70]
6			5	98 [72]
7			30	85 [73]
8			10	95 [74]
9			5	96 [64]
10			5	98 [65]
11			3	98 [66]
12			5	95 [71]
13			5	98 [40]
14			3	97 [68]

Table 1 continued

Entry	Substrate	Product	Time (min)	Yield (%)[a]
15			3	96 [54]
16			15	95 [67]
17			30	85 [75]

Amine (1 mmol), di-Boc (1.1 mmol), SBA-15-Ph-SO_3H (0.004 g), solvent-free

[a] Isolated yield

catalyst was kept for recovery. EtOH was distilled off under vacuum to yield the highly pure *N*-Boc derivative.

Results and discussion

In continuation of our interest in use of solid acid catalysts in organic transformations [56–63] and our previous report on the synthesis and application of silica phenylsulfonic acid as a solid acid heterogeneous catalyst for one-pot synthesis of 2-aryl-1-arylmethyl-1*H*-1,3-benzimidazoles and bis(indolyl)methanes in water [56], we report herein the use of mesoporous silica phenylsulfonic acid (SBA-15-Ph-SO_3H) as a recyclable and heterogeneous solid acid catalyst for *N-tert*-butoxycarbonylation of various structurally different amine derivatives (Scheme 1), the results of which are summarized in Table 1. In an initial study, aniline (1 mmol) was treated with $(Boc)_2O$ (1.1 mmol) and SBA-15-Ph-SO_3H (0.004 g, 1 mol %) as a catalyst to isolate the corresponding mono *N*-Boc derivative within 2 min in 98 % yield. The efficiency of our protocol was evaluated using a variety of structurally diverse amines. This study was extended to a wide range of structurally diverse amines including open-chain, cyclic, aromatic, and heteroaromatic compounds, as well as aniline, all of which underwent reaction smoothly with $(Boc)_2O$. The general applicability of the method for synthesis of a wide variety of diverse *N*-Boc-amines was demonstrated (Table 1, entries 1–16).

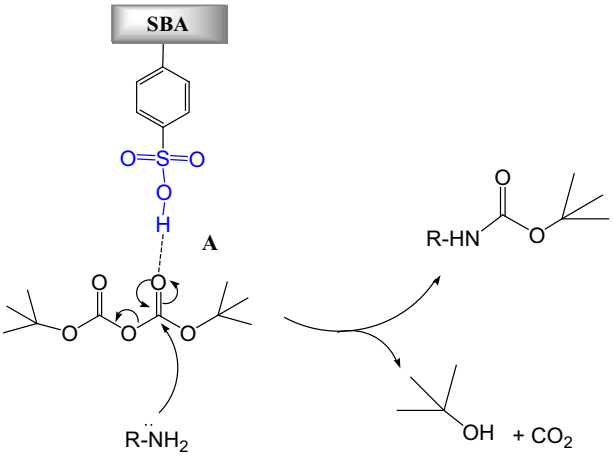
Alkylamine (cyclic and acyclic) primary and secondary amines (Table 1, entries 9–16) reacted faster and gave mono-protected derivatives in good yield in 2–10 min. In contrast, analogous reactions of primary and secondary arylamines proceeded in a sluggish manner. These results are not surprising, because alkylamines are more nucleophilic than arylamines. It is important to note that in the case of primary amines any side reaction, there were never any bis-BOC

derivatives observed, as confirmed by ^{1}H nuclear magnetic resonance (NMR) analysis of the crude products (Table 1, entries 1–4, 6-9, 12, 13, 16, 17).

With arylamines, the reaction rate was mainly dependent on the nature of substituent groups as well as their position on an aromatic ring. In general, aromatic amines with electron-withdrawing substituent reacted slower than those with electron-donating substituent (Table 1, entries 1–8). The amine group was exclusively protected in comparatively good yields even in the presence of phenolic-OH and thiophenol (Table 1, entries 4, 8) or alcohol (Table 1, entry 12) groups. Excellent chemoselectivity was observed for substrates with OH and SH functionalities, providing *N*-Boc derivatives as the major products with no significant *O*/*S*-*tert*-butoxycarbonylation taking place. This encouraged us to study competitive *N*-Boc protection of alanine as an amino acid in the presence of SBA-15-Ph-SO_3H under solvent-free condition (Table 1, entry 17). The corresponding *N*-Boc derivative of amino acid was obtained in good yield.

The catalyst can catalyze by electrophilic activation of $(Boc)_2O$ to form **A**, making the carbonyl group susceptible to nucleophilic attack by the amine. This facilitates extrusion of *tert*-butanol and carbon dioxide as leaving entities, eventually leading to formation of *N*-Boc-protected amine and catalyst (Scheme 2). The insolubility of the catalyst in organic solvents allows for easy separation of the product by simple filtration; SBA-15-Ph-SO_3H was reused without decrease in its activity.

For practical applications of heterogeneous systems, catalyst recovery is an important aspect. The reusability of the catalyst in the reaction of aniline and $(Boc)_2O$ under solvent-free condition at room temperature in 2 min was studied. In this procedure, after completion of each reaction, ethanol was added to the reaction mixture and shaken and centrifuged for a few minutes to dissolve the product. Then, the clear liquid was separated, and the residue was dried at 50 °C. A new reaction



Scheme 2 The purposed mechanism for the protection of amines with di-Boc using SBA-15-Ph-SO_3H as catalyst

was then performed with fresh aniline under the same condition. The catalyst could be reused at least 10 times without any change in its activity (Fig. 1).

Furthermore, structural elucidation of SBA-15-Ph-SO_3H was performed in some detail using transmission electron microscopy (TEM) and X-ray diffraction (XRD) techniques after use for five runs. TEM images and XRD results for the catalyst (Figs. 2, 3) showed an ordered mesostructure in large domains with no distinct defects observed for these reaction conditions. Three well-resolved diffraction peaks in the 2θ range of 0.8–2° were observed for SBA-15-Ph-SO_3H as an organic–inorganic hybrid material, like the SBA-15 parent. However, the ordered structure of SBA-15-Ph-SO_3H remained intact, as supported by the XRD results. The patterns

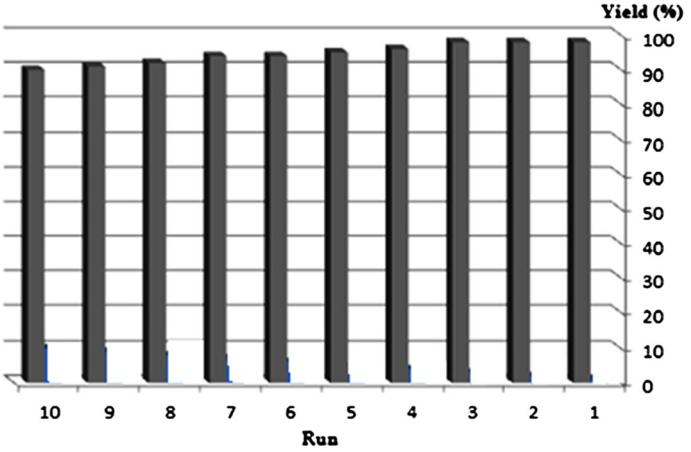


Fig. 1 Catalytic activity of SBA-15-Ph-SO_3H in 10 cycles for *N*-Boc protection of aniline

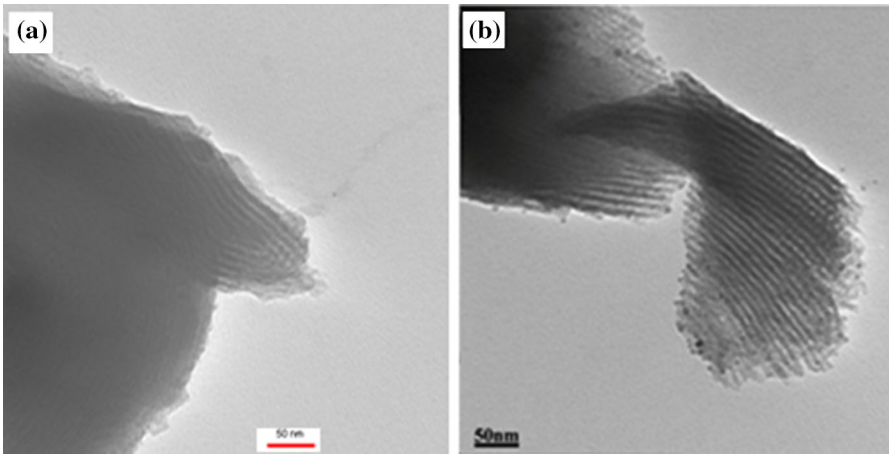


Fig. 2 TEM images of SBA-15-Ph-SO_3H: **a** fresh, **b** after use in five runs

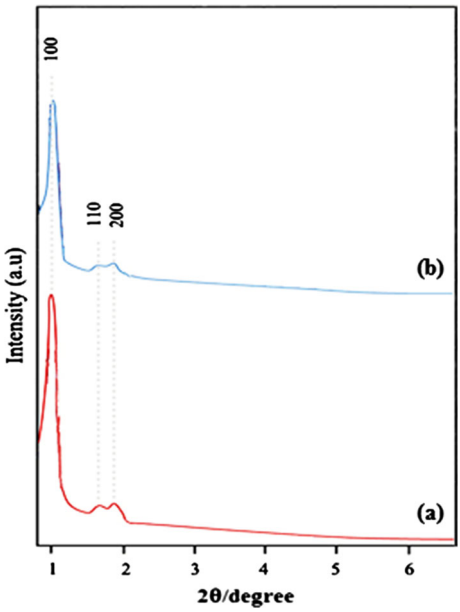


Fig. 3 XRD spectrum of SBA-15-Ph-SO3H: **a** fresh, **b** after use in five runs

Table 2 Performance comparison of various catalysts in *N*-Boc protection of aniline

Entry	Catalyst/condition	Time	Solvent	Yield	Reference
1	Cyclodextrin, r.t.	2.5 h	H_2O	75	[76]
2	Uncatalyzed, r.t.	48 h	–	60	[77]
3	[Dsim]HSO_4, r.t.	15 min	EtOH	90	[55]
4	Iodine, r.t.	30 min	Neat	95	[51]
5	Thiourea, 60 °C	40	Toluene	95	[78]
6	[H_2-cryptand 222]$(Br_3)_2$	5 h	–	80	[79]
7	SBA-15-Ph-SO_3H, r.t.	2 min	–	98	This work

feature distinct Bragg peaks in the 2θ range of 0.8–2°, which can be indexed to (1 0 0), (1 1 0), and (2 0 0) reflections of a two-dimensional hexagonal structure of SBA-15 material. The presence of these peaks indicates that the crystallographic ordering of the mesopores in SBA-15-Ph-SO_3H was retained after its use as a catalyst in five runs.

Furthermore, we compared the effect of our catalyst with reported catalysts for the reaction of aniline with di-*Boc* (Table 2). The results showed that SBA-15-Ph-SO_3H was more effective than the reported catalysts.

Conclusions

We have introduced phenylsulfonic acid-functionalized mesoporous SBA-15 silica (SBA-15-Ph-SO_3H) as a hydrophobic and recyclable nanoreactor solid acid catalyst for protection of various amines to *N*-Boc derivatives at room temperature with excellent yields. This new method offers the following competitive advantages: (i) mild and operationally simple, (ii) high activity and good chemoselectivity, (iii) no side reactions, (iv) wide substrate scope and generality in the presence of various other functions, and (v) easy workup and solvent-free nature, making the protocol environmentally benign.

Acknowledgments The authors are grateful to Iran National Science Foundation (INSF) and Payame Noor University (PNU) for supporting this work under research grant no. 93/48910 dated 2014/12/03.

Conflict of interest The authors declare that they have no conflict of interest.

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