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Silica-bonded *N*-propyl sulfamic acid as an efficient catalyst for the formylation and acetylation of alcohols and amines under heterogeneous conditions

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ABSTRACT

A simple and efficient procedure for the preparation of silica-bonded *N*-propyl sulfamic acid (**SBNPSA**) by the reaction of 3-aminopropylsilica (**1**) and chlorosulfonic acid in chloroform is described. This solid acid is employed as a new catalyst for the formylation of alcohols and amines with ethyl formate under mild and heterogeneous conditions at room temperature with good to excellent yields. Also, **SBNPSA** catalyzed acetylation of various alcohols and amines with acetic anhydride at room temperature.

1. Introduction

In recent years, the search for environmentally benign chemical processes or methodologies has received much attention.[1] Heterogenization of homogeneous catalysts has been an interesting area of research from an industrial point of view; this combines the advantages of homogeneous catalysts (high activity, selectivity, etc.) with the engineering advantages of heterogeneous catalysts (easy catalyst separation, long catalytic life, easy catalyst regeneration, thermal stability, and recyclability).[2] Application of solid acids in organic transformations is important, because, solid acids have many advantages such as simplicity in handling, decreased reactor and plant corrosion problems, and more environmentally safe disposal.[3–7]

Protection of functional groups plays an important role in the synthesis of complex organic molecules such as natural products. Formylation and acetylation have found increasing attention as highly familiar protocols for the protection of functional groups in this context.

O-Formylation could be the method of choice for protecting an alcoholic group in a complex synthetic sequence because deformylation can be affected selectively in the presence of acetate or other ester protecting groups. Further, if the alcoholic group is planned to be oxidized later in the synthetic scheme, the formylated alcoholic group need not be deprotected and direct oxidation under Oppenauer conditions can be realized.[8–11] Although various formylating agents have been reported previously, there are still serious limitations for the preparation of formates due to the drastic reaction conditions, the use of uncommon reagents, formation of undesirable or toxic by-products, the application of expensive catalysts for preparation of formylating agents, and thermal instability of the reagents.[12] To the best of our knowledge, one of the most common formylating agents is formic acid which is corrosive and toxic. Meanwhile, acid-sensitive functional groups may be affected and side reactions may be increased in the formylation reaction by using formic acid. Among formylating agents ethyl formate offers several advantages such as easy work-up, availability of the reagent, and relatively low cost. Some of the recently reported methods include formylation with ethyl formate in the presence of different reagents or catalysts, namely metal triflates such as $Ce(OTf)_4$,[13] $In(OTf)_3$,[14] Bi(III) salts,[15] heteropoly acids,[16] silphos $[PCl_{3-n}(SiO_2)_n]$,[10] silica sulfuric acid and $Al(HSO_4)_3$,[17] cerium polyoxometalate,[18] silica triflate,[19] and $TiCl_3(OTf)$.[20]

Acetylation of alcohols, phenols, and thiols is usually carried out by using acid anhydrides or acyl chlorides in the presence of protic

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acids,[21] various Lewis acid catalysts, or basic reagents such as 4-(dimethylamino)pyridine DMAP,[22,23] tributylphosphine,[24] and $La(NO_3)_3{\cdot}6H_2O$.[25] Performing the reaction in the presence of acidic catalysts has become more popular and some of the recently reported methods include applying metal triflates such as cerium triflate,[26] gadolinium triflate,[27] aluminum triflate,[28] scandium triflate,[29,30] and $Sc(Tf_2)_3$,[31] metal halides such as $TaCl_5$,[32] $InCl_3$,[33] and $ZrOCl_2{\cdot}8H_2O$,[34] and solid acid catalysts such as heteropoly acids,[35] $Mg(NTf_2)_2$,[36] aluminum-supported MoO_3,[37] cation-exchanged montmorillonite K-10 clay,[38] sulfamic acid,[39] $NaHSO_4{\cdot}SiO_2$,[40] phosphomolybdic acid,[41] polymer-supported gadolinium triflate,[42] polystyrene-bound tetrafluorophenylbis(triflyl)-methane,[43] $BiFeO_3$,[44] heterogeneous cobalt(II) salen,[45] and [TMBSA][HSO_4] ionic liquid.[46]

2. Results and discussion

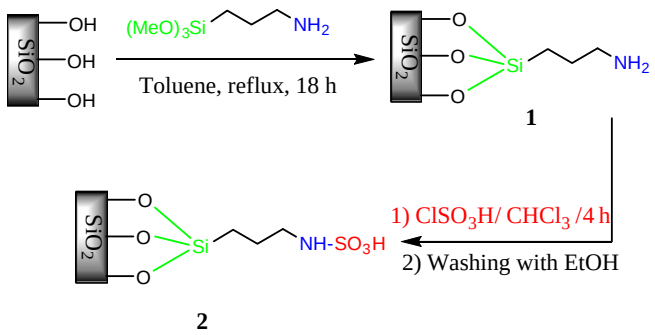
In the continuation of our studies on the applications of solid acid catalysts in chemical transformations,[47–50] herein, we wish to describe the preparation of new silica-bonded *N*-propyl sulfamic acid (**SBNPSA**) as illustrated in Scheme 1, and its use as a catalyst for the formylation and acetylation of alcohols. Elemental analysis showed the S content to be 9.29%. Typically a loading of ca. 0.35 mmol/g was obtained. When **SBNPSA** was placed in aqueous NaCl solution, the pH of the solution dropped almost instantaneously to ≈1.87, as ion exchange occurred between the protons and sodium ions (proton exchange capacity: 0.34 mmol/g of **SBNPSA**).

2.1. Formylation

As we mentioned in the introduction, owing to the easy deprotection of the formyl group, costs, and the work-up, formylation is regarded as an attractive option for the protection of different functional groups. Therefore, we investigated the formylation of alcohols with ethyl formate in the presence of **SBNPSA** as a catalyst. First, the transesterification reaction of benzyl alcohol (1 mmol) with ethyl formate (3 mL) was chosen as a model reaction. As shown in Table 1, in the absence of catalyst benzyl alcohol remains intact even after 24 h. However, in the presence of sulfamic acid (1 mmol), benzyl formate was afforded in only 45% yield after 6 h at room temperature (Table 1, entry 2). Then, we examined this reaction in the presence of different catalyst loadings of **SBNPSA** at room temperature (Table 1). The optimal amount of **SBNPSA** was 0.1 g (3.4 mol %) per 1 mmol of alcohols and 3 mL of ethyl formate at room temperature.

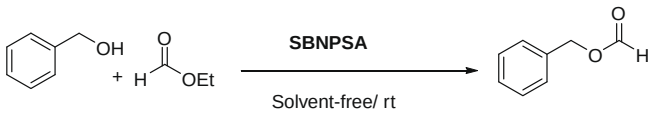
The optimized conditions (0.1 g of **SBNPSA**, and 3 mL of ethyl formate) were then used in the formylation of various alcohols under mild, nearly neutral and heterogeneous conditions (Table 2 and Scheme 2).

As shown in Table 2, primary alcohols reacted with ethyl formate faster than secondary and tertiary alcohols in the presence



Scheme 1. Preparation of silica-bonded *N*-propyl sulfamic acid (**2**).

Table 1
Influence of the amounts of **SBNPSA** on the formylation of benzyl alcohol with ethyl formate at room temperature[a]



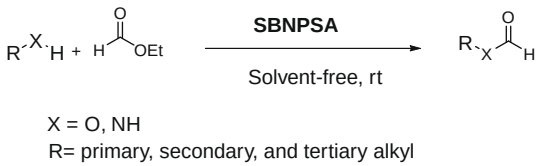
Entry	Catalyst	The amount of catalyst (g)	Time (min)	Yield[b] (%)
1	None	None	24 h	0
2	Sulfamic acid	1 mmol	360	45
3	**SBNPSA**	0.01	360	32
4	**SBNPSA**	0.015	240	51
5	**SBNPSA**	0.05	100	72
6	**SBNPSA**	0.1	45	91
7	**SBNPSA**	0.15	45	92

[a] The reaction conditions: alcohol (1 mmol) and ethyl formate (3 mL) at room temperature and under neat conditions.
[b] Isolated yield.

Table 2
Formylation of alcohols and amines with ethyl formate in the presence of **SBNPSA** at room temperature[a]

Entry	Substrate	Time (min)	Yields[b] (%)
1	Benzyl alcohol	45, 45,[c] 50,[c] 50[c]	91, 90,[c] 88,[c] 89[c]
2	4-Chlorobenzyl alcohol	80	93
3	2-Chlorobenzyl alcohol	90	89
4	4-Bromobenzyl alcohol	80	90
5	4-Methoxybenzyl alcohol	30	94
6	4-*t*-Butylbenzyl alcohol	40	92
7	4-Nitrobenzyl alcohol	120	35
8	*n*-Octanol	70	80
9	2-Phenylethanol	40	91
10	3-Phenylpropanol	45	89
11	1-Phenylethanol	40, 40,[c] 50,[c] 120[c]	91, 90,[c] 90,[c] 88[c]
12	Indanol	70	87
13	Cyclohexanol	120	78
14	1-Phenyl-2-methyl-2-propanol	24 h	30
15	Phenol	180	—[d]
16	*p*-Cresol	180	—[d]
17	Aniline	40, 40,[c] 45,[c] 45[c]	95, 93,[c] 95,[c] 90[c]
18	Aniline	24 h[e]	80[e]
19	4-Nitroaniline	8 h (24 h)[e]	40 (—)[d,e]
20	4-Methylaniline	40 (24 h)[e]	85 (80)[e]
21	4-Bromoaniline	45 (24 h)[e]	85 (77)[e]
22	Benzylamine	5 (180)[e]	90 (89)[e]
23	4-Methoxybenzylamine	5 (180)[e]	90 (88)[e]

[a] The reaction conditions: substrate (1 mmol), ethyl formate (3 mL), and catalyst 0.1 g (3.4 mol %) at room temperature and under neat conditions.
[b] Isolated yield.
[c] The reaction was accomplished with reused catalyst.
[d] No reaction.
[e] The reaction was accomplished without catalyst.



Scheme 2. Conversion of alcohols and amines into corresponding formate esters and formamides with ethyl formate in the presence of **SBNPSA**.

of **SBNPSA** at room temperature. Electron-donating substituents on the aromatic ring of benzyl alcohols, such as *para*-methoxybenzyl alcohol, reacted faster than benzyl alcohol (Table 2, entry 5), while formylation of *para*-nitrobenzyl alcohol was sluggish and the corresponding ester was isolated in only 35% yield after

120 min at room temperature (Table 2, entry 7). Our investigation clarified that benzyl amines and aniline derivatives reacted with ethyl formate in short reaction times and very good to excellent yields at room temperature. However, anilines with electron-withdrawing groups such as NO_2 required longer reaction times and were low yielding (Table 2, entry 18). It is important to note that amines reacted with ethyl formate without any catalyst in long reaction times and only near 90% conversion (Table 2, entries 18–23).

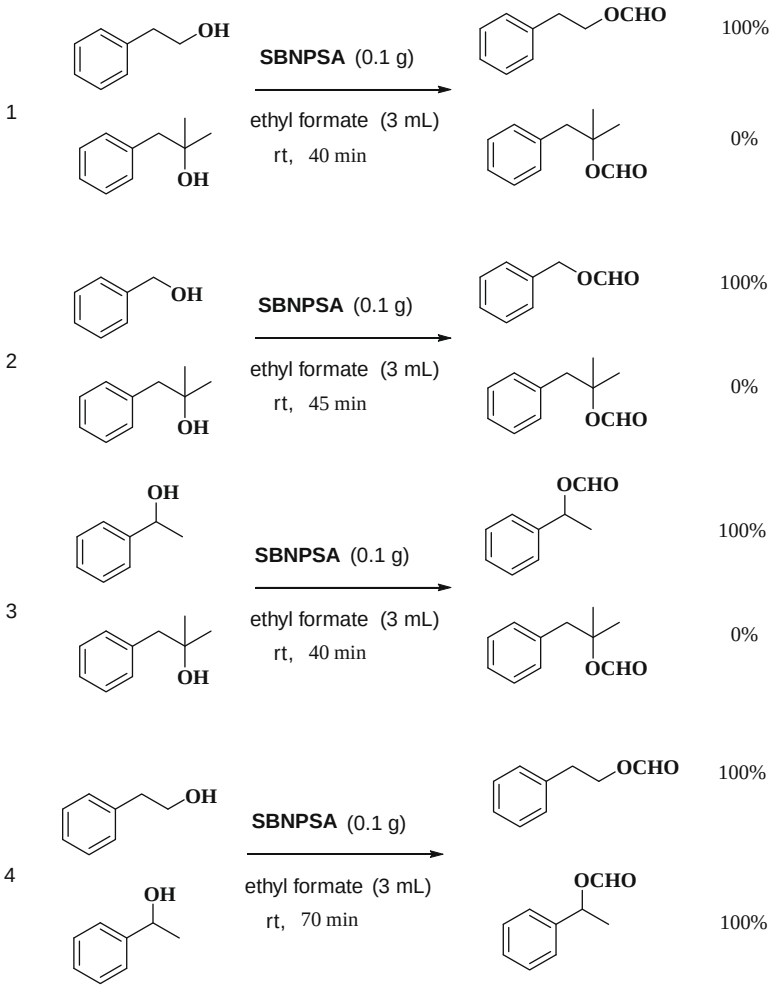
The described catalytic formylating systems are chemoselective so that formylation of alcohols occurred selectively in the presence of phenols. In addition, the selective formylation of primary or secondary alcohols in the presence of tertiary alcohols was investigated. This method was shown to be highly selective for the primary alcohols such as benzyl alcohol and 2-phenylethanol. The primary alcohols were completely converted to the corresponding formate ester, while tertiary alcohols were left untouched (Scheme 3). Excellent chemoselectivity was also observed for the conversion of secondary alcohols in the presence of tertiary alcohols such as 1-phenyl-2-methyl-2-propanol. No selectivity was observed between primary and secondary alcohols such as 2-phenylethanol and 1-phenylethanol (Scheme 3).

The possibility of recycling the catalyst was examined using the reaction of benzyl alcohol, 1-phenylethanol, and aniline with ethyl formate under optimized conditions. Upon completion, the reaction mixture was filtered and the solid was washed with diethyl ether, and the recycled catalyst was saved for next reaction. The recycled catalyst could be reused thrice without any treatment. No observation of any appreciable loss in the catalytic activity of **SBNPSA** was observed (Table 2, entries 1, 11, and 17).

2.2. Acetylation

Acetylation of alcohols is usually achieved with acetic anhydride. Acetyl is the most common protecting group in view of being stable under acidic conditions, and being easily removable by mild alkaline hydrolysis. The poor nucleophilic properties of hydroxyl compounds necessitate activation of the anhydride. Silica-bonded *N*-propyl sulfamic acid has proved to be efficient catalyst for this purpose at room temperature. The optimal amount of the **SBNPSA** was 0.1 g (3.4 mol %) per 1 mmol of benzyl alcohol and 1 mL of acetic anhydride. As illustrated in Table 3, a range of benzylic, aliphatic primary, secondary, and tertiary alcohols were acetylated with acetic anhydride in the presence of **SBNPSA** in short reaction times at room temperature (Scheme 4).

Acetylation of *ortho*- and *para*-nitrobenzyl alcohols was fast and the corresponding esters were isolated in high yields (89% and 91% yields) after 60 min, respectively (Table 3, entries 6 and 7). Phenols were also, acetylated in short reaction times with acetic anhydride in the presence of **SBNPSA** (Table 3, entries 14–17). It is important to note that according to the previous report[39] most of these acylation reactions proceeded at 62–80 °C in the presence of sulfamic acid. Amines were acetylated with acetic anhydride in the presence of **SBNPSA** or without any catalyst in short reaction times in excellent yields (Table 3, entries 18–24).

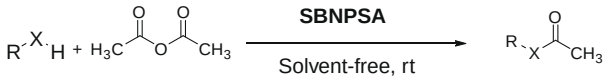


Scheme 3. Reaction conditions: each substrate (1 mmol), ethyl formate 3 mL, and catalyst **SBNPSA** (3.4 mol %) at room temperature (the yields referred to GC yield).

Table 3
Acetylation of alcohols, phenols, and amines in the presence of **SBNPSA** with acetic anhydride at room temperature[a]

Entry	Substrate	Time (min)	Yields[b] (%)
1	Benzyl alcohol	20, 20,[c] 20,[c] 25,[c] 25,[c] 28,[c] 30,[c] 30[c]	98, 94,[c] 96,[c] 95,[c] 91,[c] 92,[c] 90,[c] 90[c]
2	4-Chlorobenzyl alcohol	30	93
3	4-Methoxybenzyl alcohol	20	93
4	4-*t*-Butylbenzyl alcohol	25	91
5	3-Phenoxybenzyl alcohol	30	90
6	4-Nitrobenzyl alcohol	60	91
7	2-Nitrobenzyl alcohol	60	89
8	2-Phenylethanol	25	90
9	1-Phenylethanol	30, 30,[c] 30,[c] 30,[c] 45[c]	90, 90,[c] 90,[c] 88,[c] 88[c]
10	Indanol	30	93
11	Cyclohexanol	45	86
12	Benzhydrol	20	91
13	Triphenylcarbinol	260	90
14	Phenol	360	92
15	*p*-Cresol	340	90
16	4-Bromophenol	350	91
17	2-Naphthol	350	90
18	Aniline	10, 10,[c] 10,[c] 10,[c] 10,[c]	95, 93,[c] 92,[c] 93,[c] 91[c]
19	Aniline	5[d]	93[d]
20	4-Nitroaniline	20 (15)[d]	90 (91)[d]
21	4-Methylaniline	10 (5)[d]	95 (93)[d]
22	4-Bromoaniline	10 (5)[d]	91 (89)[d]
23	Benzylamine	15 (5)[d]	93 (90)[d]
24	4-Methoxybenzylamine	15 (5)[d]	88 (89)[d]

[a] The reaction conditions: substrate (1 mmol), acetic anhydride (1 mL), and catalyst 0.1 g (3.4 mol%) at room temperature.
[b] Isolated yield.
[c] The reaction was accomplished with reused catalyst.
[d] The reaction was accomplished without catalyst.



X = O, NH
R= primary, secondary, and tertiary alkyl

Scheme 4. Acetylation of alcohols, phenols, and amines with acetic anhydride in the presence of **SBNPSA**.

The recycling potential of the catalyst was investigated in the reaction of benzyl alcohol, 1-phenylethanol, and aniline with acetic anhydride at room temperature in the presence of 0.1 g of **SBNPSA**. After completion of the reaction, diethyl ether was added and the mixture was filtered to separate the catalyst. The recycled catalyst was used in further runs. No decrease in catalytic activity of **SBNPSA** was observed even after five runs (Table 3, entries 1, 9, and 18).

3. Conclusion

In conclusion, chemoselectivity, the cheapness and availability of the reagents, nearly neutral and heterogeneous conditions, easy and clean work-up, and high yields make this method practical for multi-step synthesis. We believe that the present methodology could be an important addition to the existing methodologies.

4. Experimental

4.1. General

Chemicals were purchased from Fluka, Merck, and Aldrich Chemical Companies. The yields of all volatile formylated products have been determined by GC. The formylated products were characterized by comparison of their spectral and physical data with previously reported data or with the authentic samples.[16–42] 3-Aminopropylsilica 1 (AMPS) was prepared according to previously reported procedure.[3]

4.2. Catalyst preparation

4.2.1. Silica-bonded *N*-propyl sulfamic acid 2

To a mixture of 3-aminopropylsilica **1** (5 g) in chloroform (20 mL) chlorosulfonic acid (1 g, 0.6 mL) was added dropwise at 0 °C over 2 h. After addition was complete, the mixture was stirred for 2 h until HCl gas evolution stopped. Then, the mixture was filtered and washed with ethanol (30 mL) and dried at room temperature to obtain silica-bonded N-propyl sulfamic acid (**2**) as a cream powder (5.13 g). Sulfur content of the samples determined by conventional elemental analysis was 9.29%.

4.3. General procedure for formylation of alcohols with ethyl formate

Solid acid **SBNPSA** (0.1 g) was added to ethyl formate (3 mL) and the mixture was stirred for several minutes. Then the substrate (1 mmol) was added to the above-mentioned mixture and the suspension was stirred at room temperature for the specified time given in Table 2. The progress of the reaction was monitored by TLC or GC. Upon completion of the reaction, the suspension was filtered off and the solid was washed with diethyl ether (2 × 10 mL). The organic phases were combined and passed through a short column of silica gel to give the pure products.

4.4. General procedure for acetylation with acetic anhydride

To a mixture of substrate (1 mmol) and acetic anhydride (1 mL), was added **SBNPSA** (0.1 g) and the mixture was stirred at room temperature for the specified time given in Table 3. The progress of the reaction was monitored by TLC or GC. When the reaction was complete, 15 mL of diethyl ether was added and the mixture was filtered, the solid was washed with diethyl ether (2 × 10 mL). The combined organic phases were washed with

10% solution of sodium hydrogen carbonate, and twice with water (2×10 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated to afford the crude product. If further purification was needed it was passed through a short column of silica gel.

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