Synthesis and Application of Modified Silica Sulfuric Acid as a Solid Acid Heterogeneous Catalyst in Michael Addition Reactions

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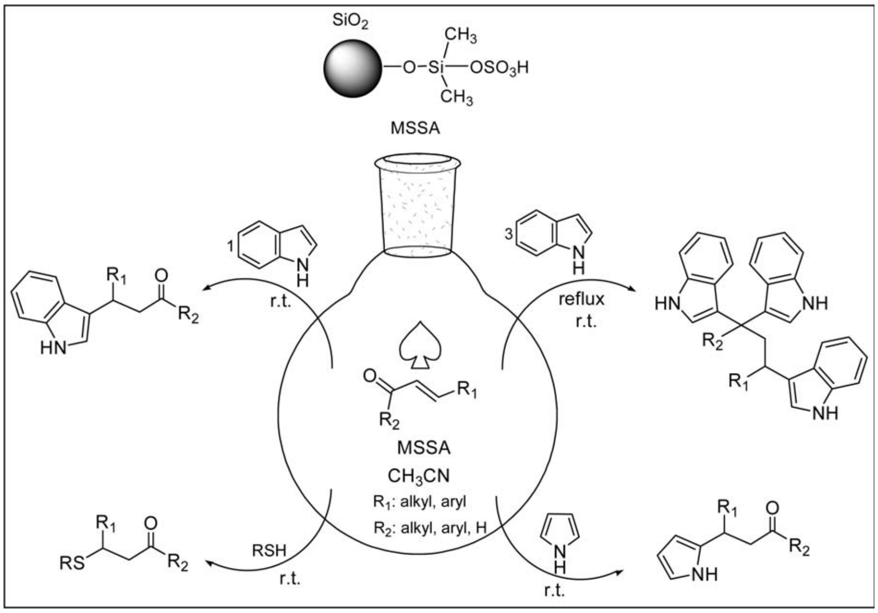

Modified silica sulfuric acid (MSSA) as a new type of silica sulfuric acid was prepared and effectively used in the conjugate addition of indole, pyrrole, and thiols with Michael acceptors under mild conditions at room temperature. Also, MSSA was used as a catalyst for the synthesis of 1,1,3-tri-indolyl compounds in good to excellent yield at room temperature.

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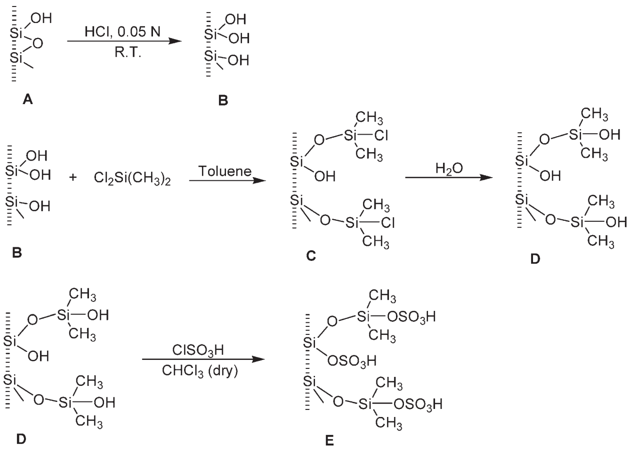
INTRODUCTION

Michael additions promoted by Lewis acid catalysts are one of the most important carbon–carbon bond-forming reactions in organic synthesis. Addition reactions of indoles, amines, and thiols to α,β-unsaturated compounds have received much interest due to a number of their derivatives occur in nature and possess a variety of biological activities [1,2]. Thus, the development of facile and environmentally friendly synthetic methods for the preparation of these compounds constitutes an active area of investigation in pharmaceutical and organic synthesis. The Michael addition is one of the most useful carbon–carbon bond-forming reactions and has wide synthetic applications in organic synthesis [3]. This reaction is traditionally catalyzed by strong bases that often lead to undesirable side reactions and/or products [4]. A variety of Lewis acids are found to catalyze this reaction, and these procedures are also not free from disadvantages [5]. Thus, a number of milder reagents and Lewis acids catalysts such as Al_2O_3 [6], K_2CO_3 [7], rhodium complex [8], ruthenium complex [9], clay-supported nickel bromide [10], quaternary ammonium salt [11], $InBr_3$ [12], $InCl_3$ [13], $Bi(NO_3)_3$ [14], I_2 [15], $Bi(OTf)_3$ [16], $HClO_4/SiO_2$ [17], GaI_3 [18], PTSA [19], sulfamic acid [20], Ru(III) [21], NH_4Cl [22], and *N*-phenyltris(dimethylamino)iminophosphorane immobilized on polystyrene resin [23] have been developed over the past few years. Indeedly, a number of these procedures have one or the other disadvantages such as longer reaction time, use of excessive

Scheme 1



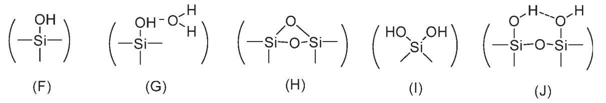
expensive catalyst, harsh reaction conditions, failure to provide addition product, and tedious experimental procedure. Consequently, there is a need for a catalytically efficient method for these transformations, which might work under mild and more economical conditions.

Any reduction in the amount of sulfuric acid needed and/or any simplification in handling procedures is required for risk reduction, economic advantage, and environment protection. In addition, during the recent years, the use of reusable heterogeneous catalysts has received considerable importance in organic synthesis because of their environmental, economical, and industrial aspects. The development of efficient methods using recoverable and reusable catalysts is an important goal in organic synthesis. Very recently, we have published a comprehensive review related of the synthesis and applications of bis- and tris-indolylmethanes [24].

RESULTS AND DISCUSSION

In continuation of our studies on the synthesis [25] and application of silica sulfuric acid (SSA) [25–29] as a solid acid heterogeneous catalyst, we wish to reported the synthesis of modified silica sulfuric acid (MSSA) as a new type of SSA in organic reaction. Although our first report of SSA has been widely used and cited by international researchers [25], we think that MSSA is superior due to its more stability in the presence of moisture. MSSA is easily prepared from the silanization of activated silica gel with dimethyldichlorosilane (DMCS) followed by treatment with water and then adding chlorosulfonic acid at room temperature (Scheme 1).

Scheme 2



The extent to which chemical reactions occur with a given porous' silica surface are controlled by the types and reactivity of chemical groups present and the steric availability of these groups. Much disagreement and confusion has arisen over these points. Earlier workers suggested the existence of three distinct groups: silanol or "bound water" (**F**), silanol with physically adsorbed water (**G**), and dehydrated oxides (**H**) [30,31]. More recently, slightly different views of the surface have arisen. Snyder [32,33] has viewed the silica surface as containing varying proportions of five groups: geminal (**I**), bound and reactive (**J**), free (**F**), and siloxane (**H**). The amount of each of these groups is dependent on structural considerations (Scheme 2).

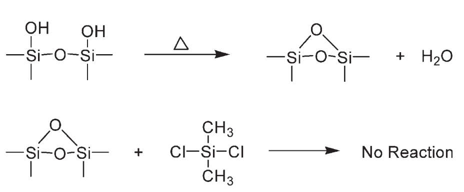
The silica surface is ordinarily considered to be covered with a monolayer of silanol groups, which arise from the tendency of each silicon atom on the surface to maintain tetrahedral coordination [34].

The dehydration of silica has been also a much argued topic. A number of authors [31,35] have suggested the following: below 150°C loss of physically adsorbed water; around 150 to 600°C evolution of bound water without appreciable structural deformation; and above 600°C, an increased possibility of internal alterations. On the other hand, Unger [36] has recently shown evidence to indicate that the hydroxyl group, silanol, concentration decreases only slightly up to 300°C, and that a pronounced decrease in silanol concentration occurs between about 300 and 500°C. This decrease is attributed to condensation of bound or paired hydroxyl groups. The condensation of free hydroxyl groups occurs only above 600°C.

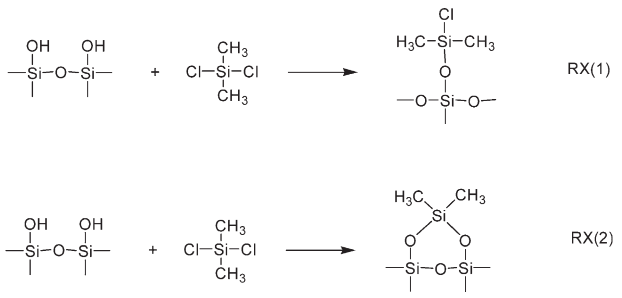
When the silica surface is heated above 150°C, partial removal of the "bound water" occurs, very slowly up to ~300 to 350°C at which point a pronounced decrease occurs to about 500 to 550°C, leaving a dehydrated oxide condition, which will not be chemically active to DMCS. These conditions may be described by the following reaction (Scheme 3).

Thus, by controlling the prereaction dehydration, varying amounts of DMCS may be chemically bonded to the porous silica surface. It has been suggested in the literature [37,38] that two possibilities exist for the reaction of DMCS with the silica surface. These possibilities are illustrated in the following equations (Scheme 4).

Scheme 3



Scheme 4



If reaction **1** is the reaction path, then the chlorine and carbon bound to the surface should have a 1: 2 ratio and be directly related to the number of OH groups available for reaction. In the event that reaction **2** is the controlling process, no chlorine will be detectable, and the ratio of bound carbon to initially available hydroxyl groups should have a one to one correspondence. However, in view of these reactions, a third complicating alternative must be considered, the possibility that both reactions **1** and **2** occur simultaneously to some degree. In this event, the number of C1 and OH groups neither show direct agreement nor the surface be completely devoid of C1. Also, the bound carbon would not show a 2:l or 1:l ratio with initially available hydroxyl groups as in the case of purely reactions **1** or **2**, respectively, but some intermediate value [39].

Thus, after activation of silica gel surface with HCl (0.05 *N*), to avoid the reaction of DMCS with adjacent hydroxyl groups to form a dimmer, we used excess amount of DMCS in reaction medium (Scheme 1). Then, for conversion of Si—Cl to Si—OH groups after silanization and drying, we added water to solid product, and stirring it for 10 min at room temperature. The amount of Cl groups in this step was determined by titration with $AgNO_3$ or with ion selective electrode (ISE). After completion of all reactions, samples were dried in a dry flowing nitrogen stream at 120°C for 24 h to maintain controlled moisture conditions. In continuation, reaction of silanol groups with chlorosulfonic acid at room temperature was caring out. It is interesting to note that the reaction is easy and clean without any work-up procedure, because HCl gas is evolved from the reaction vessel immediately (Scheme 1).

A study of the stability of bonded sulfonic acid surfaces in SSA and MSSA toward water was made. We added 1 g of SSA and MSSA in the separate column and then washed them with 20-mL water. After washing, we removed the solid and on titration of the sulfonic-coated silica surface with a solution of NaOH (0.1 *N*). The surface-bonded sulfonic acid in SSA was reduced to 1 s of MSSA. This would indicate that the stability of MSSA as a solid acid heterogeneous catalyst toward water and hydrolysis condition. This stability may be because of steric effects of methyl groups in MSSA toward water attack.

As a part of our ongoing research program to develop new synthetic methodologies [40–42], we found that MSSA as a stable solid acid heterogeneous catalyst, nonvolatile, inexpensive, and safe reagent for conjugate addition of indoles, pyrroles, and thiols to Michael acceptors under solvent conditions (Scheme 5).

First, we studied the reaction of chalcon **1a** with indole (1:1 molar ratios) to optimize the reaction conditions with respect to temperature, time, and the ratio of MSSA to the substrate. We found that 200 mg of MSSA was sufficient to obtain the desired Michael adduct in 98% yield within 90 min at room temperature in CH_3CN using chalcon **1a** (Table 1, Entry 1).

Under the optimized reaction conditions, a variety of α,β-unsaturated ketones and electron-deficient olefins were tested in acetonitrile catalyzed by 200-mg MSSA through the reaction of indole with these Michael acceptors under mild conditions. The reactions proceeded easily, and the products were isolated with

Scheme 5

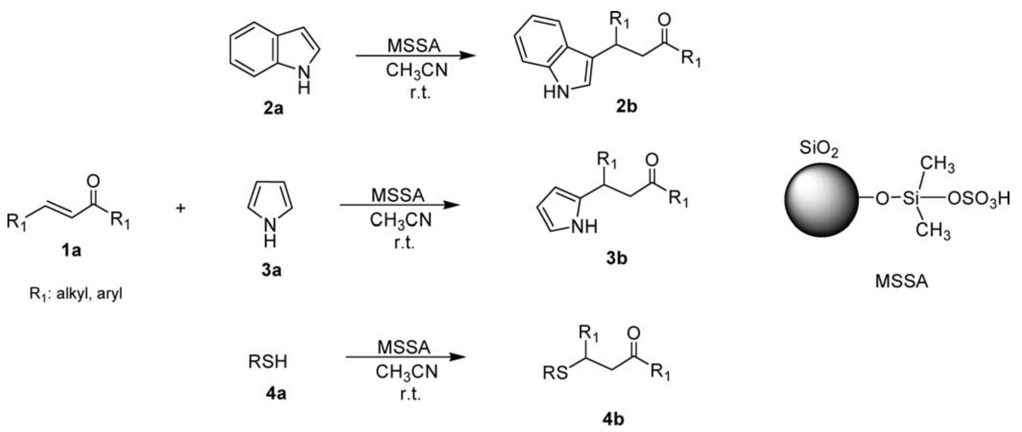


Table 1

Michael addition of indole, pyrrole, and thioles to Michael acceptors catalyzed by MSSA.

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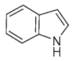
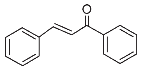
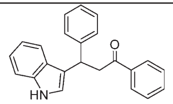
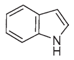
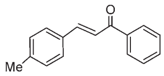
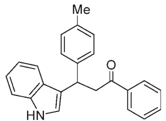
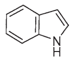
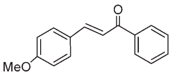
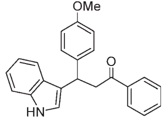
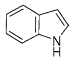
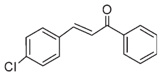
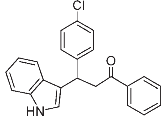
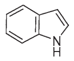
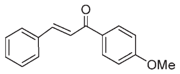
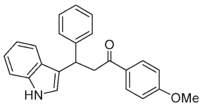
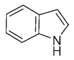
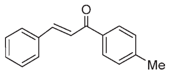
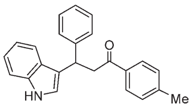
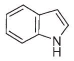
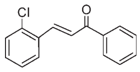
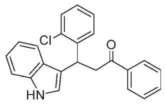
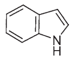
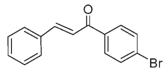
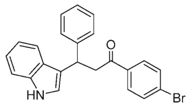
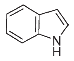
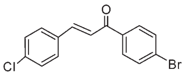
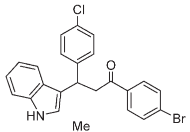
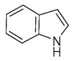
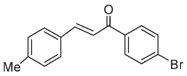
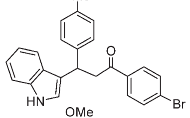
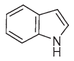
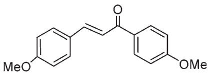
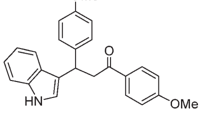
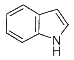
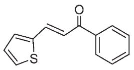
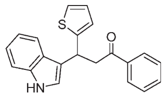
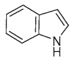
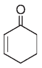
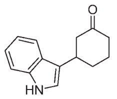
1				1.5	98	[19]
2				1.3	96	[19]
3				1.6	92	[19]
4				1.1	98	[19]
5				2	95	[48]
6				1.8	92	[19]
7				1.5	96	[46]
8				1.1	90	[46]
9				1.2	98	[41]
10				1.6	96	[41]
11				1.5	90	[48]
12				2.1	85	[19]
13				2.5	92	[16]

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Table 1
(*Continued*)

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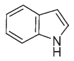
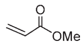
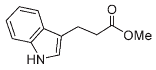
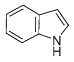
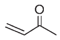
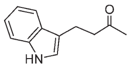
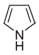
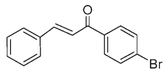
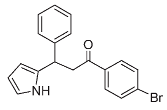
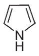
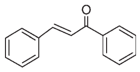
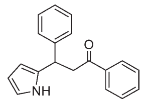
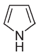
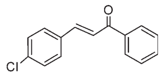
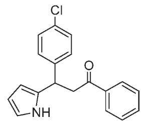
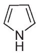
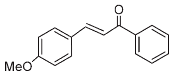
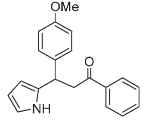
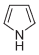
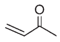
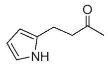
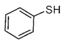
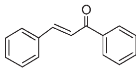
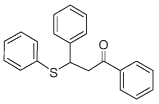
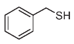
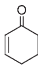
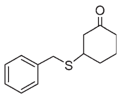
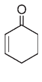
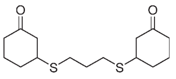
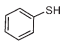
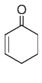
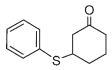
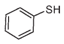
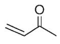
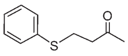
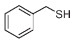
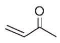
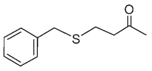
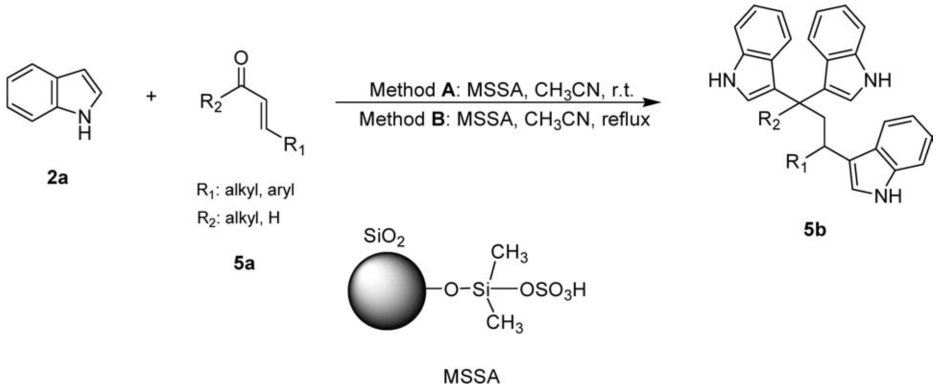
14				1.4	85	[16]
15				1.3	92	[16]
16				2	90	[41]
17				2.5	85	[47]
18				2.1	92	[41]
19				2.5	90	[47]
20				3	75	[47]
21				0.25	85	[17]
22				0.084	95	[16]
23				0.5	90	[17]
24				0.25	75	[16]
25				0.17	92	[16]
26				0.084	95	[17]

[a] Products were characterized by ^{1}H NMR, ^{13}C NMR, IR, and comparison with reported data.
[b] Isolated yields.

Scheme 6



comparable yields in short reaction times. The results are summarized in Table 1 (entries 1–15). The reactions were clean, and the products were obtained in high yields without the formation of any side products, such as *N*-alkylation product.

Apparently, the pyrrole Michael addition was also done in the same fashion as indole Michael addition. Based on the results obtained, the reaction was extended to pyrrole and was found that MSSA can also efficiently catalyzed the reaction of pyrrole with different α,β-unsaturated ketones, affording 2-substituted pyrrole derivatives with comparable yields (Table 1, entries 16–20).

Thia-Michael addition products of α,β-unsaturated carbonyl compounds are very important building blocks for the synthesis of bioactive compounds [43], heterocycles [44], and are also used as chiral auxiliary for the synthesis of optically active α-hydroxy aldehydes [45]. Therefore, the development of an efficient and selective catalyst for the construction of carbon–sulfur bond is of interest in organic synthesis. In continuation of our ongoing research program to develop better and newer synthetic methodologies [24–29,40–42], we perceived that MSSA might be a very useful catalyst for thia-Michael reaction (Scheme 5).

In a set of initial experiments, 1,3-diphenylpropenone was allowed to react with thiophenol in an equimolar ratio in the presence of a varied quantity of MSSA and various conditions. After a series of experimentations, it was observed that excellent yield of the Michael adduct can be achieved by reacting a mixture of 1,3-diphenylpropenone (1 equiv.), thiophenol (1.1 equiv.), and MSSA (100 mg) in acetonitrile (3 mL) at room temperature. Aliphatic and aromatic thiols can be used in the optimized procedure with numerous cyclic and acyclic α,β-unsaturated carbonyl compounds. Products were isolated with comparable yields, and the results were summarized in Table 1 (entries, 21–26). It was noticed that the reactions of thiols with enones yielded the desired thia-Michael addition products rapidly (5–30 min) and in an excellent yields (75–95%).

On the basis of the results of above study, we planned to synthesis of 1,1,3-triindolyl compounds using MSSA as a solid acid heterogeneous catalyst under room temperature and reflux conditions through the tandem Michael addition and Friedel–Crafts reaction of α,β-unsaturated aldehydes or ketones and indoles (Scheme 6).

In initial experiments, indole **2a** and crotonaldehyde (**5a**) were used as model substrates to evaluate suitable reaction conditions for the preparation of tris-indole **5b** (Table 2). The reaction to form 1,1,3-tri(1H-indol-3-yl)butane (**5b**) in 96–98% yield was completed in 10 h at room temperature and 20 min at reflux condition in CH_3CN when 250-mg MSSA was used as the catalyst (Table 2, entry 1).

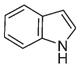
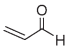
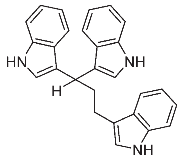
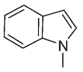
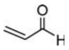
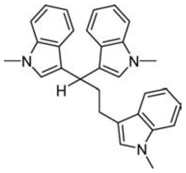
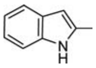
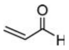
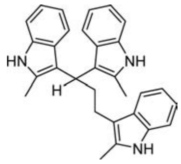
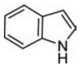
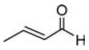
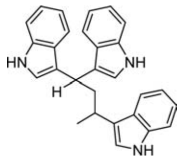
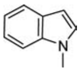
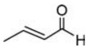
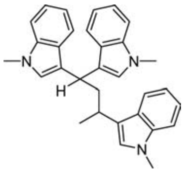
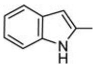
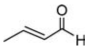
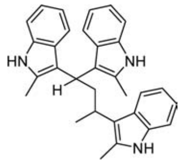
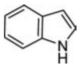
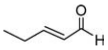
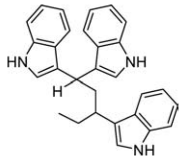
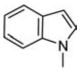
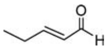
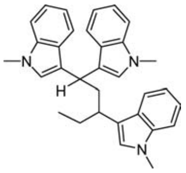
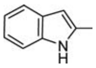
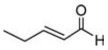
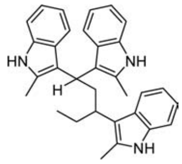
Encouraged by this initial success, the reactions were then performed between a variety of α,β-unsaturated compounds with different indoles under similar conditions, at room and reflux temperature. The results summarized in Table 2 highlight the general applicability of this reaction.

The mechanism for this three indole incorporating reaction is shown in Scheme 7. The 1,4- and 1,2-additions of indole to α,β-unsaturated ketone take place sequentially to give intermediate **K** in the presence of MSSA (Scheme 7). The dehydration of **K** gives another intermediate **L**, which is further activated by MSSA and serves as an electrophile to react with a third molecule of indole, affording intermediate **M**. The corresponding three indole-incorporated product is subsequently formed from intermediate **M**. It is thought that MSSA promotes the reaction by increasing the electrophilic character of the enal.

The reusability of the catalysts is one of the most important benefits and makes them useful for commercial applications. Therefore, we investigated the recovery and reusability of MSSA catalyst. The catalyst can be easily separated by simple filtration and reused after washing with anhydrous $CHCl_3$ and drying at 60°C. The reusability of the catalyst was checked in the Michael

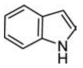
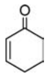
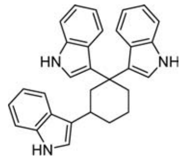
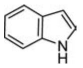
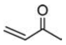
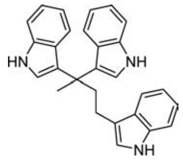
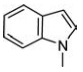
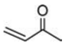
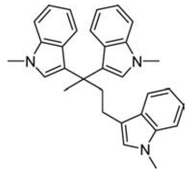
Table 2

Synthesis of 1,1,3-triindolyl compounds catalyzed by MSSA.

Entries	Nucleophile	Electrophile	Products[a]	Method A Time (h)	Method A Yield (%)	Method B Time (min)	Method B Yield (%)	Refs.
1				10	96	20	98	[43]
2				13	85	60	96	[43]
3				12.5	80	60	92	[43]
4				12	92	35	96	[43]
5				10	90	30	98	[43]
6				12	75	65	96	[43]
7				10	80	45	98	[43]
8				10	75	50	98	[43]
9				12	70	75	97	[43]

(*Continued*)

Table 2
(*Continued*)

				Method A		Method B		
Entries	Nucleophile	Electrophile	Products[a]	Time (h)	Yield (%)	Time (min)	Yield (%)	Refs.
10				15	65	90	90	[43]
11				24	25	150	75	[43]
12				24	35	120	75	[43]

addition of chalcon **1a** with indole in the presence of MSSA. As can be seen, the catalyst could be used at least four successive times without any decrease in its activity (Table 3).

CONCLUSION

In conclusion, MSSA as a new type of SSA and as a good proton source was prepared. Also, we have developed an efficient and cost-effective method for Michael addition of indole, pyrroles, and thiols to Michael acceptors and preparation of 1,1,3-triindolyl compounds in excellent yields through the tandem Michael addition and Friedel-Crafts reaction of α,β-unsaturated aldehydes or ketones, in the presence of MSSA in CH_3CN. This method has advantages, such as ease of workup, mild condition, high yields, and short reaction time and is a novel method for the synthesis of important 3-substituted indole derivatives.

EXPERIMENTAL

All commercially available chemicals were obtained from Merck and Fluka companies, and used without further purifications unless otherwise stated. ^{1}H NMR spectra were recorded on a Jeol 90 MHz FT NMR spectrometer using TMS as internal standard, and chemical shifts are in δ (ppm). Infrared (IR)

Scheme 7

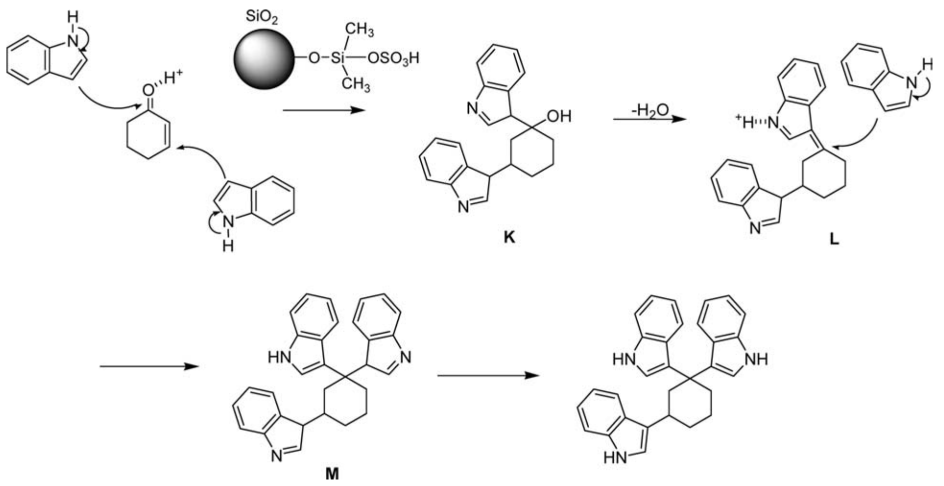


Table 3

Reusability of MSSA catalyst in the Michael addition of chalcon **1a** with indole.

Run	Time (h)	Yield (%)
1	1.5	98
2	1.5	96
3	1.5	90
4	1.5	80

was conducted on a Perkin Elmer GX FT-IR spectrometer. All yields refer to isolated products.

Preparation of MSSA. Fifty gram of silica gel **A** (SiO_2, mesh 35–70, 675 m^2/gr, Merk) was added to 150-mL HCl (0.05 *M*) in a suitable vessel and stirred for 30 min at room temperature. After 30 min, the solid was filtrated and washed with deionized water for several times to get the activated silica gel (**B**). The activated SiO_2 (**B**) was dried under vacuum (60 mm/Hg) in 70°C for 3 h.

Activated SiO_2 (50 g) was dispersed in dry toluene (70 mL) under nitrogen condition and then DMCS (50 g) was added to the dispersion and stirred for 6 h. After that, filterated and washed with dry toluene (100 mL) and Et_2O (150 mL) to get the chlorodimethysililated silica (**C**). Next step for conversion of Si—Cl (**C**) to Si—OH (**D**) groups, 200-mL water was added to the reaction vessel of **C** for 10 min stirred at room temperature. Afterward, the solid was separated by filtration and washed by 100-mL water and 100-mL acetone and dried it in vacuum at 120°C for 5 h. In the final step, 30-g dry silanizated silica (**D**) was added to solution of chlorosulfonic acid (15 g) and 40-mL dry $CHCl_3$ in a round-bottom flask at room temperature for 60 min. The flask was equipped with a gas inlet tube or $CaCl_2$ trap for conducting HCl gas. HCl gas evolved from the reaction vessel immediately. After completion of the reaction, the MSSA (**E**) was filtrated, washed with 150-mL $CHCl_3$ and 150-mL Et_2O, and dried under vacuum (60 mm/Hg) in 80°C. A white solid (MSSA) of 41.38 gr was obtained.

The Michael addition of indole and pyrrole to α,β-unsaturated carbonyl compounds in the presence of MSSA as a solid acid heterogeneous catalyst. A mixture of indole or pyrrole (1 mmol), α,β-unsaturated carbonyl compound (1 mmol), MSSA (200 mg), and CH_3CN (3 mL) was stirred at room temperature for the appropriate time (Table 1). After completion of the reaction (monitored by TLC, hexane/acetone 3:1), the reaction mixture was diluted with CH_3CN (10 mL) and filtrated. Evaporation of the solvent followed by recrystallization from ethanol–water (70:30) afforded pure Michael adducts, which were characterized by spectral methods.

General procedure for the Michael addition of thiols to α,β-unsaturated carbonyl compounds in the presence of MSSA as a solid acid heterogeneous catalyst. To a mixture of thiol (1.1 mmol), α,β-unsaturated carbonyl compound (1 mmol) in CH_3CN (3 mL) and MSSA (100 mg) was added. The mixture was allowed to stir at room temperature for a period time specified in Table 1. The reaction was monitored by TLC (3:1 *n*-hexane/acetone). After completion of the reaction, the reaction mixture was diluted with CH_3CN (10 mL) and filtrated. Evaporation of the solvent followed by short column chromatography over silica gel (petroleum ether/ethyl acetate, 95:5, v/v) afforded pure thia-Michael adducts, which were characterized by spectral methods.

General procedure for the preparation of 1,1,3-triindolyl compounds using MSSA as a solid acid heterogeneous catalyst. To a stirring solution of α,β-unsaturated carbonyl compound (1 mmol) and indole (4 mmol) in CH_3CN (5 mL), MSSA (200 mg) was added at room temperature or reflux conditions. After completion of the reaction and filtration, the solvent was evaporated under reduced pressure. The product was purified by flash column chromatography using *n*-hexane-ethyl acetate (8:2) as eluent to give pure adducts.

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