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PREPARATION OF SILICA-BONDED S-SULFONIC ACID: A RECYCLABLE CATALYST FOR THE SYNTHESIS OF BIS-INDOLYLMETHANES

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Silica bonded S-sulfonic acid (SBSSA) was prepared by the reaction of 3-mercaptopropylsilica (MPS) and chlorosulfonic acid in chloroform. Silica-bonded S-sulfonic acid, as a reusable solid acid catalyst, was used for the condensation reactions of indole with carbonyl compounds at ambient temperature. Tetra-(indolyl)methanes were also obtained by the condensation reaction of indole with dialdehyde compounds. Silica bonded S-sulfonic acid was recovered and reused.

Supplemental materials are available for this article. Go to the publisher's online edition of Phosphorus, Sulfur, and Silicon and the Related Elements to view the free supplemental file.

Keywords Bis(indolyl)methanes; carbonyl compounds; indole; silica-bonded S-sulfonic acid; tetra(indolyl)methanes

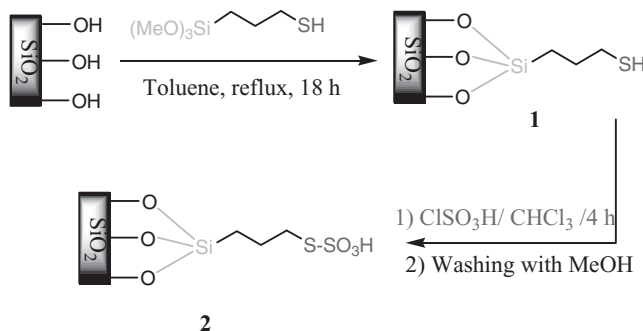
INTRODUCTION

The development of heterogeneous catalysts for fine chemical synthesis has become a major area of research, as the potential advantages of these materials, such as simplified recovery and reusability, the potential for incorporation in continuous reactors and microreactors, etc., over homogeneous systems can lead to novel, environmentally benign chemical procedures for academia and industry.¹ Application of solid acids in organic transformation have an important role because solid acids have many advantages such as simplicity in handling, decreased reactor and plant corrosion problems, and increased environmentally safe disposal.^{2–12} It is clear that green chemistry not only requires the use of environmentally benign reagents and solvents, but also it is very crucial to recover and reuse the catalyst. One way to overcome the problem of recyclability of traditional acid catalysts is to chemically anchor their reactive center onto a large surface area inorganic solid carrier to create

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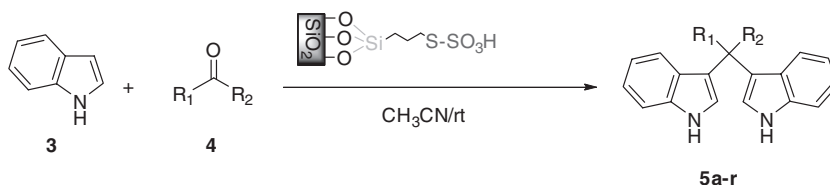


Scheme 1 Preparation of silica-bonded S-sulfonic acid (SBSSA).

a new organic–inorganic hybrid catalyst.^{4–6} In these types of solids, the reactive centers are highly mobile, similar to homogeneous catalysts, and at the same time they have the advantage of recyclability like heterogeneous catalysts. In this view, several types of solid sulfonic acid functionalized silica (both amorphous and ordered) have been synthesized and applied as an alternative to traditional sulfonic acid resins and homogeneous acids in catalyzing chemical transformations.^{5–7} Along the line of our studies in application of solid acid catalysts in chemical transformations,^{8–12} in this article we wish to describe the preparation of silica bonded S-sulfonic acid (SBSSA), as illustrated in Scheme 1 and its use as a catalyst in the condensation reaction of indole with carbonyl compounds under mild conditions.

Indole and its derivatives are important intermediates in organic synthesis and exhibit various physiological properties and pharmacological activities.¹³ Over the past decade, a number of natural products containing bis(indolyl)methanes or bis(indolyl)ethanes have been isolated from marine sources.¹⁴ Bis(indolyl)methanes, which are found in cruciferous plants and are known to promote beneficial estrogen metabolism,¹⁵ induce apoptosis in human cancer cells. Therefore the preparation of these intermediates has received increased attention from synthetic organic chemists and biologists. Generally, this reaction is promoted using protic acids,¹⁶ Lewis acids,¹⁷ heteropolyacid,¹⁸ and heterogeneous catalysts (clay, HY-zeolites, and supported reagents).^{19,20} Recently, sulfamic acid,²¹ silica sulfuric acid,²² $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ /silica gel,²³ ionic liquids in conjunction with $\text{In}(\text{OTf})_3$ or $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$,²⁴ HClO_4 - SiO_2 ,²⁵ $\text{M}(\text{HSO}_4)_n$,²⁶ ammonium chloride,²⁷ SbCl_3 ,²⁸ silica chloride,²⁹ heteropoly acid in water,³⁰ $\text{P}_2\text{O}_5/\text{SiO}_2$,³¹ $\text{Zr}(\text{DS})_4$,³² ZrCl_4 ,³³ iodine,³⁴ PEG-supported sulfonic acid,³⁵ and ZnO ³⁶ were employed for this transformation. Although the methods mentioned above are suitable for the reaction described, there sometimes exist some drawbacks such as long reaction time, expensive reagents, low yield of products in some cases, high catalyst loading, corrosive reagents, and large amounts of solid supports, which would eventually result in the generation of a large amount of toxic waste. For this reason, superior catalysts that are cheap, less toxic, easily available, air-stable, recyclable, and water-tolerant, are desirable.

In connection with our interest in the use of solid acids as efficient catalysts in various transformations, and in continuation of our studies on the preparation of indolylmethanes,^{22a,26,31} in this article we report the preparation of bis(indolyl)- and tetra(indolyl)methanes from indole and carbonyl compounds in the presence of silica bonded SBSSA at room temperature (Scheme 2).



Scheme 2 Silica-bonded S-sulfonic acid-catalyzed condensation of carbonyl compounds with indole.

RESULTS AND DISCUSSIONS

First, we tried to condense indole (1 mmol) with benzaldehyde (0.5 mmol) and silica-bonded S-sulfonic acid (0.1 g) in the presence of various solvents (Table I). As shown in Table I, the best result was obtained in acetonitrile in terms of time and product yield.

The model reaction was also examined in the presence of various amounts of SBSSA, and as shown in Table II, the best result was obtained as the following: indole (1 mmol), aldehyde (0.5 mmol), and SBSSA (0.1 g) in acetonitrile at room temperature.

These results prompted us to investigate the scope and the generality of this new protocol for various aldehydes and ketones under optimized conditions (Table III). A series of aromatic aldehydes and simple ketones smoothly underwent an electrophilic substitution reaction with indole to afford a wide range of substituted bis(indolyl)methanes in good to excellent yields. This method is equally effective for aldehydes bearing electron-withdrawing or -donating substituents in the aromatic rings. Furthermore, acid-sensitive aldehydes worked well without any decomposition or polymerization under these reaction conditions.

In all cases, aldehydes reacted more rapidly and gave higher yields than ketones. Most of the reactions of aromatic aldehydes proceeded nearly quantitatively, and the nature of substituents on the aromatic ring showed some effects on this conversion. In other words, the electron-donating substituents such as CH_3 and OCH_3 required a shorter reaction time than the electron-deficient counterparts such as CN and NO_2 . Ketones required longer reaction times and lower yields, which is most probably due to the steric effects of the methyl group (Table III, entries 15–16).

The possibility of recycling the catalyst was examined using the reaction of indole (10 mmol) with benzaldehyde (5 mmol) in acetonitrile (10 mL), in the presence of SBSSA

Table I The effect of solvents on the conversion of indole (1 mmol) and benzaldehyde (0.5 mmol) into phenyl-3,3'-diindolylmethane in the presence of SBSSA (0.1 g) at room temperature

Entry	Solvent	Time (min)	Yield (%) ^a
1	H ₂ O	120	—
2	CH ₃ CN	30	100
3	CH ₃ COOC ₂ H ₅	120	75
4	CH ₂ Cl ₂	120	85
5	CH ₃ OH	60	95
6	C ₂ H ₅ OH	60	96
8	THF	90	95
9	Chlorobenzene	120	55

^aConversion.

Table II The condensation of indole with benzaldehyde catalyzed by SBSSA in acetonitrile at room temperature

Entry	The amount of catalyst (g)	Time (min)	Yield (%) ^a
1	0.01	90	75
2	0.03	75	80
3	0.05	60	90
4	0.07	40	95
5	0.1	30	100

^aConversion.

(0.5 g) at room temperature. Upon completion, the reaction mixture was filtered, the remaining solid was washed with ethanol, and the catalyst was reused in the next reaction. The recycled catalyst could be reused 19 times without any additional treatment. No observation of any appreciable loss in the catalytic activity of SBSSA was observed (Figure S4, Supplemental Materials).

This reaction was further explored for the synthesis of tetra-indolyl compounds **5q**, **5r** by the condensation of terephthalaldehyde **6** and 2,6-diformyl-4-methoxyphenol **7** with four equivalent of indol under similar conditions in very good yields (Scheme S1, Supplemental Materials; Table III, entries 17, 18).

To show the efficiency of the SBSSA in comparison with previously reported procedures in the literature, Table IV compares some of our results with silica sulfuric acid,^{22b} PEG-supported sulfonic acid,³⁵ HY-zeolite,²⁰ ZrOCl₂·8H₂O/ silica gel,²³ sulfamic acid,²¹ and ZnO (VI)³⁶ with respect to reaction times and yields of obtained products. Also, the reusability of SBSSA catalyst is good (19 times) in comparison with the previously reported literature based on silica or polymer supported catalysts.

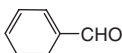
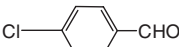
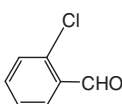
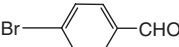
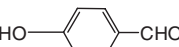
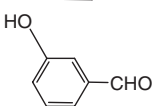

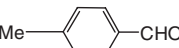
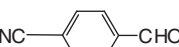
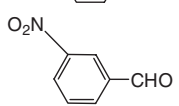
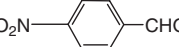
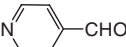
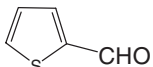
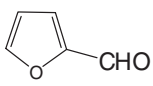
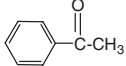

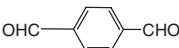
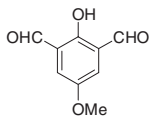
CONCLUSION

In conclusion, SBSSA has been employed for the synthesis of bis(indolyl)-methanes and tetra(indolyl)methanes by the electrophilic substitution reaction of indole with aldehydes or ketones. The attractive features of this procedure are the mild reaction conditions, high conversions, cleaner reaction profiles, and reusable and environmentally friendly catalyst, all of which lead to an interesting strategy for the preparation of bis(indolyl)methanes and tetra(indolyl)methanes.

EXPERIMENTAL

Chemicals were purchased from Merck, Fluka, and Aldrich chemical companies, and 2,6-diformyl-4-methoxyphenol was prepared according to our previously reported procedure.³⁷ 3-Mercaptopropylsilica 1 (MPS) was prepared according to the previously reported method.⁵ IR spectra were run on a Shimadzu Infrared Spectroscopy FT-IR 8000. The ¹H NMR and ¹³C NMR were run on a Bruker Avance DPX 250 MHz and 300 MHz instruments. Melting points were recorded on a Melting Point SMP1 apparatus in open capillary tubes and are uncorrected. The progress of reaction was followed with TLC using silica gel SILG/UV 254 plates. All of the products are known compounds and are

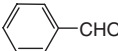
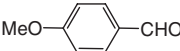
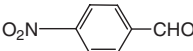
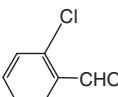
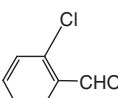
Table III SBSSA-catalyzed condensation of indole with various carbonyl compounds to produce corresponding bis(indolyl)methane at room temperature^a

Entry	Carbonyl compound	Product	Time (min)	Yield (%) ^b
1		5a ³⁶	30	95
2		5b ³⁶	60	91
3		5c ³⁶	90	90
4		5d ³⁶	60	82
5		5e ³¹	90	80
6		5f ³³	90	82
7		5g ³⁸	25	93
8		5h ²¹	30	90
9		5i ²¹	90	89
10		5j ³⁸	90	88
11		5k ³⁶	90	91
12		5l ³⁶	120	90
13		5m ²¹	120	89
14		5n ³⁵	120	87
15		5o ³⁸	360	65
16		5p ²¹	270	79
17		5q ^{22a}	210	89
18		5r ^{22a}	210	83

^aReaction conditions: indole (2 mmol), carbonyl compound (1 mmol), and SBSSA (0.1 g) in 2 mL of CH₃CN at room temperature.

^bIsolated yield.

Table IV Comparison some of the results of SBSSA^a with silica sulfuric acid^b (I),^{22b} PEG-supported sulfonic acid^c (II),³⁵ HY-zeolite^d (III),²⁰ ZrOCl₂·8H₂O/ silica gel^e (IV),²³ sulfamic acid^f (V),²¹ and ZnO^g (VI)³⁶ in the preparation of bis(indolyl)methanes at room temperature

Entry	Aldehyde	SBSSA	Time (min) / Yield (%)					
			I	II	III	IV	V	VI
1		30/95	40/92	150/95	60/85	40/84	20/88	45/98
2		25/93	15/94	120/90	60/78	—	15/94	90/96
3		90/91	—	480/86	60/85	30/94	—	20/95
4		90/90	—	180/85	60/82	—	—	45/95
5		60/91	30/90	210/90	60/65	—	60/90	30/95

The equivalent ratios of aldehyde/indole/catalyst are ^a1/2/0.1 g; ^b2/4/0.1 g; ^c1/2/0.45 g; ^d2.5/5/0.5 g; ^e1/2/5 mol%; ^f1/2/0.2 mmol; ^g1/2/0.1 mmol.

characterized by the comparison of their spectral (IR, ¹H NMR), TLC, and physical data with those reported in literature.

Catalyst Preparation

To a magnetically stirred mixture of 3-mercaptopropylsilica **1** (5 g) in CHCl₃ (20 mL), chlorosulfonic acid (1.00 g, 9 mmol) was added dropwise at 0°C during 2 h. After addition was complete, the mixture was stirred for 2 h until HCl was removed from the reaction vessel. Then, the mixture was filtered and washed with methanol (30 mL) and dried at room temperature to obtain silica-bonded functionalized sulfonic acid **2** (SBSSA) as cream powder (5.22 g). Sulfur content of the samples by conventional elemental analysis was 16.12%. Typically a loading at ca. 0.35 mmol/g is obtained. On the other hand, when the washed sulfonated product SBSSA was placed in an aqueous NaCl solution, the solution pH dropped virtually instantaneously to pH ≈ 1.85, as ion exchange occurred between protons and sodium ions (proton exchange capacity: 0.34 mmol/g of SBSSA), which is in good agreement with the result obtained from TGA and titration.

Catalyst Characterization

Thermal gravimetric analysis (TGA). The weight change of catalyst precursors was measured using a TGA simultaneous thermal analyzer apparatus of Mettler TA-4000 under a flow of dry air. The temperature was raised from room temperature to 600°C using a linear programmer at a heating rate of 20°C/min. The sample weight was 9 mg. The TGA curve for the catalyst is illustrated in Figure S1 (available online in the Supplemental

Materials). The thermo gravimetric curve seems to indicate two-stage decomposition, which is considered to be due to removal of physical absorbed water (80–110°C) or basic silica sulfonic acid (120–300°C), respectively, and is involved with a total overall weight loss of catalyst of 50%.

FT-IR spectrum of silica-bonded S-sulfonic acid. The FT-IR spectrum of the catalyst is shown in Figure S2 (Supplemental Materials). The catalyst is a solid, and the solid state IR spectrum was recorded using the KBr disk technique. For silica (SiO_2), the major peaks are broad antisymmetric Si–O–Si stretching from 1200 to 1000 cm^{-1} and symmetric Si–O–Si stretching near 802 cm^{-1} , and bending modes lies near 470 cm^{-1} . For sulfonic acid functional group, the FT-IR absorption range of the O=S=O asymmetric and symmetric stretching modes lies in 1176.5 and 1072.3 cm^{-1} , respectively, the S–O stretching mode lies in 550–700 cm^{-1} , and that of the S–S stretching mode lies from 400 to 500 cm^{-1} . FT-IR spectrum shows the overlap asymmetric and symmetric stretching bands of SO_2 with Si–O–Si stretching bands in the silica functionalized S-sulfonic acid. The spectrum also shows a broad OH stretching absorption from 3600 to 2491.9 cm^{-1} .

X-ray diffraction (XRD) of silica-bonded S-sulfonic acid. Powder X-ray, a diffraction measurement, was performed using D8 Advance Diffract Meter made by the Bruker Axs Company in Germany. Scans were taken with a 2θ step size of 0.05 and a counting time of 1.0s using Cu $\text{K}\alpha$ radiation source generated at 40 kV and 30 mA. Specimens for XRD were prepared by compaction into a glass-backed aluminum sample holder. Data were collected over a 2θ range from 5° to 100°, and phases were identified by matching experimental patterns to entries in the Diffract^{plus} version 6.0 indexing software (Figure S3, Supplemental Materials).

General Procedure: Synthesis of Bis-(indolyl)methanes 5

To a solution of indole **1** (2 mmol) and aldehyde or ketone (1 mmol) in acetonitrile (2 mL), SBSSA (0.1 g) was added and magnetically stirred at room temperature. The progress of the reaction was followed by TLC. After completion of the reaction, the suspended mixture was filtered. The remained catalyst was washed with ethanol (2×5 mL). The evaporation of ethanol gave the product which was purified by recrystallization from EtOAc/petroleum ether (1/2) or plate chromatography on silica gel eluted with EtOAc/petroleum ether (1/2).

General procedure, synthesis of tetra-(indolyl)methanes 5q, 5r. To a solution of indole **1** (4 mmol) and dialdehyde **6** or **7** (1 mmol) in acetonitrile (3 mL), SBSSA (0.2 g) was added and magnetically stirred at room temperature. The progress of the reaction was followed by TLC. After completion of the reaction, the suspended mixture was filtered. The remaining catalyst was washed with ethanol (2×10 mL). The evaporation of ethanol gave the product, which was purified by plate chromatography on silica gel eluted with EtOAc:petroleum ether (1:2).

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