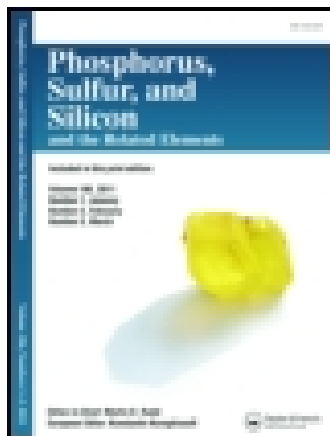


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Silica Sulfuric Acid/Wet SiO₂ as a Novel Heterogeneous System for Cleavage of Carbon Nitrogen Double Bonds Under Mild Conditions

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SILICA SULFURIC ACID/WET SiO₂ AS A NOVEL HETEROGENEOUS SYSTEM FOR CLEAVAGE OF CARBON NITROGEN DOUBLE BONDS UNDER MILD CONDITIONS

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Silica sulfuric acid in the presence of wet SiO₂ was caused to react with oximes, hydrazones, semicarbazones, azines, and Schiff-bases. It was observed that it converts them to their corresponding carbonyl compounds in good to excellent yields under mild and heterogeneous conditions.

Keywords: Silica sulfuric acid; carbon nitrogen double bond cleavage; oximes; hydrazones; semicarbazones; azines; Schiff-bases

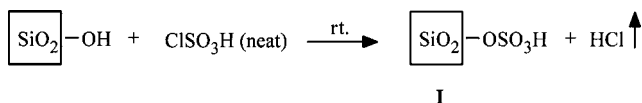
Acids are catalysts that are used most in industry for producing more than 1×10^8 Mt/year of products.¹ Among acid catalysts, the most commonly used are HF, H₂SO₄, HClO₄, and H₃PO₄ (in liquid form or supported on Kieselguhr). Solid acids have many advantages such as their simplicity in handling, likelihood of decreasing reactor and plant corrosion problems, and having environmentally safe disposal.^{1,2} Also, wastes and by-products can be minimized or avoided by developing cleaner synthesis routes.³ On the other hand, reduction in the amount

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of sulfuric acid needed and/or any simplification in handling procedures is required for risk reduction, economic advantage, and environment protection. In addition, there is current research and general interest in heterogeneous systems because of their importance in industry and developing technologies.⁴

Heterogeneous organic reactions have proven useful to chemists in the laboratory as well as in the industrial context. These reactions are effected by the reagents immobilized on the porous solid supports and have advantages over the conventional solution phase reactions because of the good dispersion of active reagent sites, associated selectivity, and easier work-up.⁵ Recently, we, among many others, have demonstrated that heterogeneous reagent systems have many advantages such as simple experimental procedures, mild reaction conditions, and minimization of chemical wastes as compared to the liquid phase counterparts.^{6,7} In continuation of our studies on the application of inorganic acidic salts and silica chloride,⁶ we found that silica gel reacts with chlorosulfonic acid to give a white powder namely silica sulfuric acid (**I**). 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).



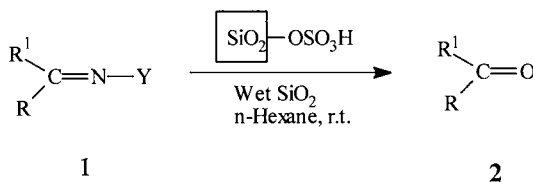
SCHEME 1

We hoped that the silica sulfuric acid (**I**) would be a superior proton source compared to all reported solid supported acids or acidic resins, such as polystyrene sulfonic acid and Nafion-H (Nafion has the handicap of low surface area which renders it unrealistic for practical use)² for running reactions under heterogeneous conditions. On developing cleaner organic reactions and also the application of this new solid acid,⁷ we were interested in using this inorganic acidic resin (**I**) for cleavage of carbon-nitrogen double bond (C=N) when used in conjunction with wet SiO₂ and *n*-hexane.

Oximes, hydrazones, azines, semicarbazones, and Schiff-bases have been employed as ketone or aldehyde functional group equivalents in organic synthesis. The regeneration of carbonyl compounds from their derivatives provides an attractive method for the synthesis of aldehydes and ketones.⁸⁻¹² Since oximes can be prepared from noncarbonyl compounds,¹³⁻¹⁸ the regeneration of carbonyl compounds from oximes provides an alternative method for the preparation of aldehydes and

ketones. Some of the methods of generating carbonyl compounds from oximes^{19–21} involve reagents that are often hazardous or very toxic, expensive, or not readily available, they need to be freshly prepared or the reactions require drastic conditions, long reaction times and tedious work-up. Thus a milder, selective, nonhazardous, and inexpensive reagent is still in demand. Although, many methods have been developed recently, for the oxidative cleavage of oximes, hydrazones, and semicarbazones to the corresponding carbonyl compounds,^{22–32} little attention has been paid to the cleavage of the carbon-nitrogen double bonds of azines and Schiff-bases. We report a simple, cheap, and heterogeneous method for the effective cleavage of carbon-nitrogen double bond (C=N) under mild conditions.

The results of the conversions of various oximes, hydrazones, azines, semicarbazones, and Schiff-bases to their corresponding carbonyl compounds are presented in Scheme 2, and Table I. The products of the reaction with silica sulfuric acid (**I**) in the presence of wet SiO₂ were isolated simply by filtering the mixture and evaporating the solvent from the filtrate. The method has advantages in terms of yields, its heterogeneous nature, cheapness and availability of the reagents, short reaction times, and easy work-up.



SCHEME 2

The oximes, hydrazones, azines, semicarbazones, and Schiff-bases were prepared by standard procedures.^{33–35} The purity of the compounds was checked by TLC and by their melting points. A mixture of the substrate, *n*-hexane, silica sulfuric acid, and wet SiO₂ was stirred at room temperature for the specified time. (The molar ratio of silica sulfuric acid (**I**), and wet SiO₂ to the substrate was optimized; see Table I), The reaction was monitored by TLC. After completion of the reaction the mixture was filtered and the solid residue was washed with *n*-hexane. Evaporation of the solvent followed by column chromatography on silica gel gave the corresponding carbonyl compounds in good to excellent yields.

In contrast to the normal Schiff-bases, which are unstable in the presence of water molecules, we synthesized a good range of Schiff-bases with a high degree of stability (Table I).³⁵ Schiff-bases **3**

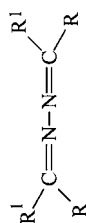
TABLE I Carbon-Nitrogen Double Bond (C=N) Cleavage with Silica Sulfuric Acid (I) and Wet SiO₂ (II) in *n*-hexane at Room Temperature

Entry	Substrate 1	Product ^a	Reagent (g/ substrate (mmol))		Time (min)	Yield ^b (%)
			I	II		
	$\begin{array}{c} \text{R}^1 \\ \diagdown \\ \text{C}=\text{N}-\text{OH} \\ \diagup \\ \text{R} \end{array}$					
	Oximes					
1	3-Nitrobenzaldehyde oxime	3-Nitrobenzaldehyde	0.8	0.8	120	75
2	4-Nitrobenzaldehyde oxime	4-Nitrobenzaldehyde	0.8	0.8	30	92
3	2-Chlorobenzaldehyde oxime	2-Chlorobenzaldehyde	0.8	0.8	60	80
4	4-Chlorobenzaldehyde oxime	4-Chlorobenzaldehyde	0.8	0.8	60	89
5	Acetophenone oxime	Acetophenone	0.8	0.8	60	82
6	4-Nitroacetophenone oxime	4-Nitroacetophenone	0.8	0.8	60	85
7	Cyclohexanone oxime	Cyclohexanone	0.8	0.8	60	74
8	Vanillin oxime	Vanillin	0.8	0.8	60	82
9	Salicylaldehyde oxime	Salicylaldehyde	0.8	0.8	60	85
10	4-Methyl-2-pentanone oxime	4-Methyl-2-pentanone	0.8	0.8	90	78
	$\begin{array}{c} \text{R}^1 \\ \diagdown \\ \text{C}=\text{N}-\text{NHCONH}_2 \\ \diagup \\ \text{R} \end{array}$					
	Semicarbazones					
11	Benzaldehyde semicarbazone	Benzaldehyde	0.8	0.8	20	100 ^c
12	Acetophenone semicarbazone	Acetophenone	1	1	35	100 ^c
13	4-Bromobenzaldehyde semicarbazone	4-Bromobenzaldehyde	1	1	10	100 ^c
14	4-Methoxybenzaldehyde semicarbazone	4-Methoxybenzaldehyde	1	1	10	100 ^c
15	3-Methoxybenzaldehyde semicarbazone	3-Methoxybenzaldehyde	1	1	15	100 ^c
16	4-Phenylacetophenone semicarbazone	4-Phenylacetophenone	1	1	5	100 ^c
17	1-Naphthaldehyde semicarbazone	1-Naphthaldehyde	1	1	10	100 ^c
18	5-Methyl-2-furaldehyde semicarbazone	5-Methyl-2-furaldehyde	1	1	10	100 ^c
19	Cyclohexanone semicarbazone	Cyclohexanone	1	1	15	100 ^c



Phenylhydrazones

20	Benzaldehyde-4-nitrophenylhydrazone	Benzaldehyde	0.8	15	100 ^c
21	4-Methylbenzaldehyde-4-nitrophenylhydrazone	4-Methylbenzaldehyde	0.8	10	100 ^c
22	4-Methylbenzaldehyde phenylhydrazone	4-Methylbenzaldehyde	0.8	50	100 ^c
23	4-Chlorobenzaldehyde phenylhydrazone	4-Chlorobenzaldehyde	0.8	40	100 ^c
24	3-Nitrobenzaldehyde phenylhydrazone	3-Nitrobenzaldehyde	0.8	300	100 ^c
25	2-Nitrobenzaldehyde phenylhydrazone	2-Nitrobenzaldehyde	0.8	175	50 ^c
26	2-Benzoylpyridine phenylhydrazone	2-Benzoylpyridine	0.8	225	100 ^c
27	2-Acetylpyridine phenylhydrazone	2-Acetylpyridine	0.8	90	100 ^c
28	4-Pyridinecarboxaldehyde phenylhydrazone	4-Pyridinecarboxaldehyde	0.8	40	100 ^c



Azines

29	Benzaldehyde azine	Benzaldehyde	0.8	60	75
30	4-Chlorobenzaldehyde azine	4-Chlorobenzaldehyde	0.8	60	90
31	4-Nitrobenzaldehyde azine	3-Nitrobenzaldehyde	0.8	60	93
32	4-Hydroxybenzaldehyde azine	4-Hydroxybenzaldehyde	0.8	60	73
33	Acetophenone azine	Acetophenone	0.8	60	78
34	4-Nitroacetophenone azine	4-Nitroacetophenone	0.8	60	86
35	2-Ethoxybenzaldehyde azine	2-Ethoxybenzaldehyde	0.8	60	80
36	2-Methyl-2-pentanone azine	2-Methyl-2-pentanone	0.8	60	79
37	Cyclohexanone azine	Cyclohexanone	0.8	60	73

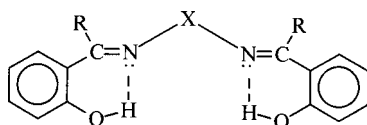
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TABLE I Carbon-Nitrogen Double Bond (C=N) Cleavage with Silica Sulfuric Acid (**I**) and Wet SiO₂ (**II**) *n*-hexane at Room Temperature (*Continued*)

Entry	Substrate I	Product	Reagent (g)/ substrate (mmol)		Time (min)	Yield ^b (%)
			I	II		
	$\begin{array}{c} \text{R}^1 \\ \diagdown \\ \text{C}=\text{N}-\text{Ar} \\ \diagup \\ \text{R} \end{array}$					
	Schiff-bases					
38	2-HO-C ₆ H ₄ -C=N-C ₆ H ₄ -2-OH	2-Hydroxybenzaldehyde	0.25	0.25	15	80
39	4-(2-HO-C ₆ H ₄ -C=N)-C ₆ H ₄ -CH ₂ -C ₆ H ₄ -4-(N=C-C ₆ H ₄ -OH)	2-Hydroxybenzaldehyde	0.5	0.5	60	80
40	2-HO-C ₆ H ₄ -C=N-(CH ₂) ₂ -N=C-C ₆ H ₄ -2-OH	2-Hydroxybenzaldehyde	0.5	0.5	25	80
41	2-HO-C ₆ H ₄ -C=N-(CH ₂) ₂ -N=C-C ₆ H ₄ -2-OH	2-Hydroxybenzaldehyde	0.5	0.5	35	82
42	2-HO-C ₆ H ₄ -C=N-(CH ₂) ₂ -NH-(CH ₂) ₂ -N=C-C ₆ H ₄ -2-OH	2-Hydroxybenzaldehyde	0.5	0.5	25	87
43	2-HO-C ₆ H ₄ -C=N-(CH ₂) ₃ -NH-(CH ₂) ₃ -N=C-C ₆ H ₄ -2-OH	2-Hydroxybenzaldehyde	0.5	0.5	20	76
44	2-HO-C ₆ H ₄ -C=N-(CH ₂) ₂ -NH-(CH ₂) ₃ -N=C-C ₆ H ₄ -2-OH	2-Hydroxybenzaldehyde	0.5	0.5	15	87
45	2-HO-C ₆ H ₄ -C=N-(CH ₂) ₂ -NH-(CH ₂) ₂ -NH-(CH ₂) ₂ -N=C-C ₆ H ₄ -2-OH	2-Hydroxybenzaldehyde	0.75	0.75	10	82
46	2-(2-HO-C ₆ H ₄ -C=N)-C ₅ H ₃ N-5-(N=C-C ₆ H ₄ -2-OH)	2-Hydroxybenzaldehyde	0.5	0.5	235	80
47	N(CH ₂) ₂ N=C-C ₆ H ₄ -2-OH) ₃	2-Hydroxybenzaldehyde	0.75	0.75	15	81

^aProducts were characterized by their physical constants, comparison with authentic samples and IR and NMR spectroscopy.^bIsolated yields.^cConversion.

are very stable due to intramolecular hydrogen bonding.^{36–37} Different kinds of Schiff-bases were also subjected to carbon-nitrogen double bond (C=N) cleavage in the presence of silica sulfuric acid (I), wet SiO₂ (50% *w/w*), and *n*-hexane as a solvent under mild and heterogeneous conditions. All of the above mentioned reactions proceeded without any application of oxidizing agents. Therefore, we think that the most interesting feature of our results is the remaining of the oxidable functional groups in the course of the reactions. While under oxidative cleavage of carbon-nitrogen double bond oxidable functional groups will be oxidized. This method is also suitable for acid sensitive functional groups due to its heterogeneous nature. Therefore, original aldehydes were obtained with excellent yields and their oxidation to the corresponding acids did not occur.



In conclusion silica sulfuric acid in the presence of wet SiO₂ can serve as an efficient system for the cleavage of the carbon-nitrogen double bond of oximes, hydrazones, azines, semicarbazones, and Schiff-bases. The yields are good to excellent and the procedure is simple and convenient. Moreover, the reaction is heterogeneous, which makes it a suitable procedure for green chemical manufacturing.³⁸

EXPERIMENTAL

General procedure: A mixture of the substrate, silica sulfuric acid and wet SiO₂ (SiO₂/H₂O: 50% *w/w*) in *n*-hexane was stirred for the time. (The molar ratio of silica sulfuric acid (I), and wet SiO₂ to the substrate was optimized; see Table I). The reaction was monitored by TLC or GLC. After completion of the reaction, the mixture was filtered. Anhydrous MgSO₄ was added to the filtrate and filtered. Evaporation of the solvent, being followed by column chromatography on silica gel [eluent: acetone/petroleum ether (1:5)], gave the corresponding carbonyl compounds in good to excellent yields.

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