

Montmorillonite K10 Catalyzed Selective Oxidation of Sulfides to Sulfoxides Using Hydrogen Peroxide

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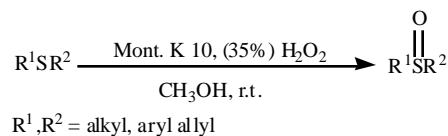
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Abstract: A clean and highly selective oxidation of sulfides to sulfoxides with 35% hydrogen peroxide catalyzed by Montmorillonite K10 in methanol is described. A series of structurally different sulfides were oxidized selectively at room temperature in excellent yields. This reaction is very simple and proceeds without over-oxidation under mild condition, and shows many advantages over the existing methodologies. Using this procedure, sulfides bearing functional groups such as hydroxyl, methoxy, amino, nitrile, aldehyde, and olefinic double bond remained intact.

Keywords: Montmorillonite K10, hydrogen peroxide, sulfide, sulfoxide, catalyzed sulfoxidation.

Convenient synthesis of organic sulfoxides is a challenging research in synthetic organic chemistry because of the importance of the sulfoxides as intermediates in chemically and biologically active compounds [1]. Oxidation of sulfides is a very useful route for preparation of sulfoxides and several oxygenating reagents as well as halogenating ones are available. This type of transformation by various oxidation methods has been extensively reviewed [2]. However, in conventional oxidation methods undesirable overoxidation to sulfone [3], and in halogenating reactions the C-S bond cleavage, formation of halo-derivatives or halogenation of aromatic ring are quite frequent competing reactions [4]. Besides the extended reaction period, most of the oxidation methods use expensive, hazardous peracids and toxic heavy metal or rare oxidant that are difficult to prepare and detract their practical application in many cases. The selective oxidation of sulfides to sulfoxides has also been developed using transition metal catalyzed sulfoxidation with hydrogen peroxide and derivatives [5], and metal Schiff base complexes in combination with a variety of oxidant have been used as catalysts for this purpose [6]. Furthermore, an enzymatic catalysis has also been reported [7]. However the major shortcoming of all heme-dependent peroxidases and Schiff base-metal complexes is their low operation stability due to facile oxidative degradation of heterocyclic or porphyrine ring. Heterogeneous catalyzed oxidative transformations by solid supports have gained popularity during the past decades thanks to distinct advantages of selectivity, mildness and operational simplicity. An increasingly popular category of supported catalysts and reagents have been developed in an effort to achieve facile, efficient, cheap and selective methods [8]. In the course of our studies on selective oxidative organic transformations [9, 10], and using solid support mediated reaction [11], and to elaborate safer and simpler oxidation process, we have investigated the role of natural clays in catalytic oxidation of sulfides to corresponding sulfoxides using hydrogen

peroxide. So far, the capability of hydrogen peroxide as an efficient oxidant has been studied considerably both from environmental and economical viewpoints. It is ideal waste avoiding oxidant and its aqueous solution shows safety in usage, storage and handling [12, 13]. But, as mentioned in the literature, without activation with a catalyst, it has to be used in controlled manner to avoid over-oxidation to sulfone [14]. Drabowicz [15] reported that for selective oxygenation of sulfides to sulfoxides by hydrogen peroxide in the absence of catalyst, the reaction time was extremely long and some other functional groups were easily destroyed. This is the first report of the clay catalyzed selective oxidation of sulfides to sulfoxides using 35% H₂O₂ as an oxygen donor, without affecting other functional groups (Scheme 1).



Scheme 1.

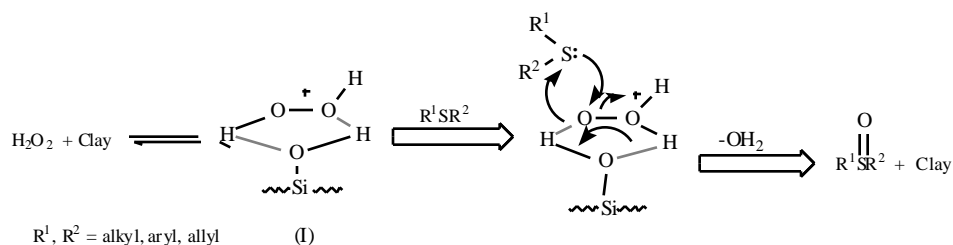
In an extended experiment to optimize the reaction conditions, the oxidation of methyl phenyl sulfide using 35% H₂O₂ in the presence of Montmorillonite K10 was attempted in different solvents. When the reaction was carried out in aprotic solvents such as diethylether, dichloromethane, n-hexane and dioxane, the unreacted methyl phenyl sulfide could be detected even after 8 hours. However, it was found that protic solvent, such as methanol, was adopted as the reaction medium since the methyl phenyl sulfoxide was produced as sole product. To explore the capability of the catalyst, we performed a series of reactions using methyl phenyl sulfide (1.0 mmol) and 35% H₂O₂ (1.0 mmol) with various type of clay catalysts (0.2 g) without any co-catalyst or promoter (Table 1). The reactions were conducted at room temperature for 3 hours by magnetic stirring at constant rate.

As evident from Table 1, the complete conversion was accomplished in the presence of small amounts of Montmorillonite K10 within 2.5 hours and methyl phenyl sulfoxide was obtained in quantitative yield. In a controlled experiment in the absence of Montmorillonite K10, it was

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Table 1. Conversion of Methyl Phenyl Sulfide to Methyl Phenyl Sulfoxide in the Presence of Clay Catalysts^a

Entry	PhSMe/ H ₂ O ₂ / Catalyst			Yield ^b (%)	Time(h)
1	1	1	Mont. K10: 0.2 g	98	2.5
2	1	1	Mont. KSF: 0.2 g	95	3
3	1	1	Bentonite: 0.2 g	60	4
4	1	1	Kaolin: 0.2 g	65	3
5	1	1	Silica gel(GF): 0.2 g	65	3
6	1	1	Englehard: 0.2 g	80	3
7	1	1	none	30	8

^aReactions were carried out in methanol at room temperature.^bGC Yields.**Scheme 2.**

found that using even a six fold excess of hydrogen peroxide, after a long reaction time (12 h), the reaction was not completed. These studies suggest that clay K10 could catalyze the oxidation of sulfide to the corresponding sulfoxide using 35% H₂O₂. As shown in Table 2, oxygenation of methyl phenyl sulfide under this reaction condition revealed that the use of 3 fold excess of hydrogen peroxide yielded sulfoxide in 1 hour, (Table 2, entry 3) without any by-product formation, and prolonging the reaction time up to 10 hours did not make any change. The reaction proceeds faster at higher reaction temperature even using a lower amount of the clay (Table 2, entry 6). The optimum ratio of sulfide/H₂O₂/clay, 1:1:0.2g was found to be ideal for complete conversion of the model compound.

Increasing the amount of clay beyond this ratio did not result in further increases in oxidation rate which demonstrating that the proton or acidic sites of clay played an important role in the reaction course. It is suggested that Montmorillonite K10 clay with high surface area can act as strong acid due to its interlayer cation and surface protons. The acid-base interaction or hydrogen bonding of clay with hydrogen peroxide would enhance the electrophilicity and reactivity of hydroperoxy intermediate (I) much higher than inactivated hydrogen peroxide. At the same time, the departing of water as a leaving group would be assisted by nucleophilic attack of sulfide (Scheme 2).

To show the general applicability of this procedure, the oxidation of a variety of structurally different sulfides was studied (Table 3). A series of substrates, aryl, alkyl, aryl alkyl, diallyl sulfides were oxidized to the corresponding sulfoxides in excellent yields. The reactivity and reaction periods were dependent on the nature of the substituents as expected. In the case of diallyl sulfide, no oxidation was observed at carbon-carbon double bond. Similarly, sulfides

bearing benzylic substituent were oxidized to the corresponding sulfoxides without affecting the active benzylic C-H bond. This clay catalyst system efficiently promotes the oxidation of aromatic and aliphatic sulfides using hydrogen peroxide in methanol at ambient temperature. Therefore, Montmorillonite K10 system was chemoselective, tolerating various functional groups including methoxy, amino, nitrile, hydroxyl and olefinic linkage. It was a significant observation that the hydroxyl and aldehyde groups were not oxidized in such oxidation condition. In order to show the chemo-selectivity of the method further, we have also investigated the competitive reactions of sulfides with sulfoxides, alcohols, aldehydes, and nitriles using this system. It is found that sulfides are converted to the corresponding sulfoxides in good to high yield with excellent selectivity.

Table 2. Optimization of the Reaction Condition in Methanol at 25°C^a

Entry	PhSMe/ H ₂ O ₂ / Montmorillonite K10			Yield (%)	Time(h)
1	1	1	0.3 g	98	2.5
2	1	2	0.3 g	98	1.5
3	1	3	0.3 g	98	1
4	1	1	0.2 g	98	2:5
5	1	1	0.15 g	90	3
6	1	1	0.15 g	95 ^a	1.5

^aReaction was performed at 50 °C.

Consequently, it is of importance to note that in spite of a number of reagents available for this transformation, the

Table 3. Selective Oxidation of Sulfides to Sulfoxides with H₂O₂ in The Presence of Montmorillonite K10^{a,b}

Entry	R ₁	R ₂	Time(h)	Yield ^c (%)	Lit(found/ ^o C)	Ref
1	Ph	Me	2:5	98	33-34 (31.5-32.5)	[17]
2	Ph	PhCH ₂	4.5	95	117-121 (116-118)	[18]
3	4-MeC ₆ H ₄	PhCH ₂	5.5	98	135-136 (136-137)	[19]
4	4-ClC ₆ H ₄	PhCH ₂	8	92	- (128-130)	-
5	4-OMeC ₆ H ₄	PhCH ₂	5	98	- (105-107)	-
6 ^d	4-NO ₂ C ₆ H ₄	Ph	15	70	100-101 (101-103)	[20]
7	2-NH ₂ C ₆ H ₄	Ph	5	98	oil	-
8	PhCH ₂	PhCH ₂	6.5	95	133-135 (134.5)	[17]
9	Ph	CH ₂ CH ₂ OH	5	98	150-152.2 (151-152)	[21]
10	Ph	CH ₂ COOH	6.5	95	112.5-113 (113-114)	[22]
11	Me	CH ₂ CH ₂ CHO	0.5	98	oil	-
12	4-ClC ₆ H ₄	CH ₂ CN	8	90	69-73 (70-71)	[9]
13	CH ₂ =CHCH ₂	CH ₂ =CHCH ₂	0.2	98	40-40.5 (40-41)	[23]
14	Cyclohexyl	PhCH ₂	2:5	98	oil	[24]
15	-CH ₂ CH ₂ CH ₂ CH ₂ -		0.2	98	105-107 (104-105)	[25]
16	n-Bu	n-Bu	2	95	32.6 (32)	[26]

^aAll reactions were carried out in methanol at room temperature using an optimized ratio of 1/1/0.2g of sulfide/ H₂O₂ /clay.

^bAll products were characterized spectroscopically (¹H NMR, IR) and showed physical and spectral data in accordance with their expected structure and by comparison with authentic samples.

^cYields refer to pure isolated products.

^dThe reaction was carried out in methanol/dioxane (1:1).

Table 4. Comparison of Our Method with Those Reported in Literature in Oxidation of Sulfides to Sulfoxides

Substrate	Reagent/Condition/Time	Yield (%)	Ref.
PhSMe	H ₂ O ₂ , MTO/ CDCl ₃ , 20 °C/24h	97	[3a]
PhSMe	PhCH ₂ Ph ₃ PHSO ₅ / CH ₃ CN, reflux/ 2h	91	[3b]
PhSMe	Sodium perborate/ CH ₃ CO ₂ H, 50-55 °C	70	[3d]
PhSMe	(Salen)Cr(III)PIO/ CH ₂ Cl ₂ , r. t./ 2h	99	[6a]
PhSMe	Ca(OCl) ₂ , wet alumina / CH ₂ Cl ₂ , r. t./40 min	90	[8b]
PhSMe	CAN, hydrated SiO ₂ / CH ₂ Cl ₂ / 1h	100	[8d]
PhSMe	Clayfen/CH ₂ Cl ₂ /18h	90	[27]
PhSMe	(Bu ₄ N)[PMo ₁₂ O ₄₀], UHP/ 4 °C/ 72h	85	[28]
PhSMe	PhIO/ PTS. (Cat)/CH ₃ CN, r. t./1h	82	[29]
PhSMe	Fe ₂ O ₃ / O ₂ , RCHO/1h	81	[30]
PhSMe	(CH ₃) ₃ COOH/H ₂ O, 0 °C	-	[31]
PhSMe	H ₂ O ₂ , Mont. K10/ CH ₃ OH, r. t./ 2.5h	98	
(n-Butyl) ₂ S	H ₂ O ₂ , MTO/ CDCl ₃ , 20 °C/24h	97	[3a]
(n-Butyl) ₂ S	PhCH ₂ Ph ₃ PHSO ₅ / CH ₃ CN, reflux/ 12h	78	[3b]
(n-Butyl) ₂ S	Sodium perborate/ CH ₃ CO ₂ H, 55 °C	93	[3d]
(n-Butyl) ₂ S	Ca(OCl) ₂ , wet alumina / CH ₂ Cl ₂ , r.t. /40 min	88	[8b]
(n-Butyl) ₂ S	CAN, hydrated SiO ₂ / CH ₂ Cl ₂ / 40 min	90	[8d]
(n-Butyl) ₂ S	Clayfen/CH ₂ Cl ₂ /12h	83	[27]
(n-Butyl) ₂ S	PhIO/ PTS. (Cat)/CH ₃ CN	91	[29]
(n-Butyl) ₂ S	Bi(NO ₃) ₃ ·5H ₂ O/ AcOH, r.t/ 2h	68	[32]
(n-Butyl) ₂ S	wet silica gel, MMPP/CH ₂ Cl ₂ /1h	99	[33]
(n-Butyl) ₂ S	(n-C ₄ H ₉) ₄ N ⁺ IO ₄ ⁻ /CHCl ₃ /2h	90	[34]
(n-Butyl) ₂ S	UHP/Ti-beta/ Acetone, 20 °C / 2h	94	[35]
(n-Butyl) ₂ S	H ₂ O ₂ , Mont. K10/CH ₃ OH, r.t./ 2.5h	95	

Magnesium Monoperxyphthalate=(MMPP); Urea hydrogen peroxide= (UHP); N,N-Ethylenebis(3,5-di-tert-butylsalicylideneiminato) chromium (III)=[(Salen)Cr(III)]; Sodium perborate tetrahydrate=(NaBO₃·4H₂O); *p*-Toluenesulfonic Acid=(PTS)

reactions either are not selective or suffer from at least one of the mentioned drawbacks. In addition, the ease of setup and workup of the reaction, and low cost and commercially availability of the oxidant and catalyst together with the excellent selectivity proposes the method to be useful for environmentally safe oxidation of sulfides to sulfoxides. In order to show the advantages of our method, we compared our results with those reported in the literature (Table 4).

EXPERIMENTAL

Chemicals and Montmorillonite K10 were purchased from Merck and Fluka,-AG chemical companies. A number of sulfides were prepared using the standard synthetic method [16]. All sulfoxides were separated and purified by different chromatographic techniques and also identified by the comparison of their m.p, IR, NMR and b.p with those reported in the literature. ¹H NMR spectra were recorded on JEOL 90 MHz NMR spectrometers. IR spectra were recorded on Pye-Unicam SP 1100 spectrophotometer.

General Procedure for Oxidation of Sulfides Using Montmorillonite K10

To a solution of sulfide (1 mmol) in methanol (4 ml), 35% H₂O₂ (1 mmol) and Montmorillonite K10 (200 mg) was added. The resulting mixture was stirred at room temperature and monitored by TLC. On completion of the reaction, the clay was removed by filtration. The solvent was evaporated and the crude sulfoxide purified on silica plate (eluent: hexane-ethylacetate, 14:6) to give excellent yield of pure product.

Oxidation of Methyl Phenyl Sulfide to Methyl- Phenyl Sulfoxide as a Typical Procedure

To a solution of methylphenylsulfide (1 mmol, 124 mg) in methanol (4 ml), H₂O₂ (2 mmol) and Montmorillonite K10 (200 mg) was added. The resulting mixture was stirred at room temperature. The reaction was monitored by TLC (n-Hexane-ethylacetate, 2/1). After completion of the reaction, the reaction mixture was filtered and the solvent was removed under reduced pressure. Recrystallization from ethanol - water, afforded pure methylphenylsulfoxide in 98% yield (Mp: 31.5-32.5 °C).

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