

Polyvinylpyrrolidone-Supported Hydrogen Peroxide (PVP-H₂O₂), Silica Sulfuric Acid and Catalytic Amounts of Ammonium Bromide as Green, Mild and Metal-Free Oxidizing Media for the Efficient Oxidation of Alcohols and Sulfides

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Polyvinylpyrrolidone-supported hydrogen peroxide (PVP-H₂O₂), silica sulfuric acid (SiO₂OSO₃H) and catalytic amounts of ammonium bromide (NH₄Br) are introduced as green media for the *in situ* generation of bromonium ion (Br⁺), which was applied for the selective oxidation of sulfides and alcohols into sulfoxides and carbonyl compounds, respectively. The oxidation reactions were carried out heterogeneously in acetonitrile, as solvent, at room temperature (oxidation of sulfides) and/or 60 °C (oxidation of alcohols).

Keywords: Polyvinylpyrrolidone-H₂O₂, Ammonium bromide, Silica sulfuric acid, Sulfide, Alcohol, Oxidation

INTRODUCTION

In the last few years, supported reagents on the organic polymers have been increasingly used in organic functional group transformations [1-4], mainly because the reactions are carried out under mild conditions and the organic products are easily isolated from the reaction media.

The controlled oxidizing of sulfides into sulfoxides is of great interest in organic chemistry. Oxidation of organo-sulfur compounds finds application in the removal of sulfur-containing pollutants during air and wastewater treatment [5]. The long-standing interest in the sulfoxidation of organic sulfides is due to their utility in diverse areas of chemistry originating from the constant use of sulfoxides as chiral auxiliaries in total synthesis, as well as useful important synthetic intermediates for the construction of various chemically and biologically active molecules [6-9]. The common synthetic procedure for the preparation of sulfoxides

is *via* oxidation of the corresponding sulfides. There are several procedures available for this key transformation, which is typically achieved using stoichiometric or catalytic amounts of both organic and inorganic reagents [10-18].

Moreover, the chemoselective oxidation of alcohols to the corresponding carbonyl compounds is an important transformation in synthetic organic chemistry, as aldehydes and ketones are essential for the preparation of many key synthetic intermediates [19-21]. Traditional methods involve the use of toxic and expensive metal oxidants [22-24], or require harsh reaction conditions [25-29].

Hydrogen peroxide (H₂O₂) is a powerful oxidizing agent. It has been used as an efficient oxidant for the oxidation of different organic functional groups [30-33]. In order to avoid the hazards connected with the use of concentrated solution of H₂O₂, this compound has been adducted with some carriers such as urea [34], DABCO-di-N-oxide [35], melamine [36] and polyvinylpyrrolidone [37]. From among these, polyvinylpyrrolidone-supported hydrogen peroxide (PVP-H₂O₂) has many advantages such as insolubility in organic

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solvents, easy work up of products, non-toxic content and mild oxidizing properties. Therefore, we decided to investigate the scope and limitations of this polymeric agent for the oxidation of sulfides and alcohols.

EXPERIMENTAL

Chemicals and Apparatus

Chemicals were purchased from Fluka, Merck and Aldrich chemical companies. Polyvinylpyrrolidone-supported hydrogen peroxide (PVP-H₂O₂) was prepared *via* the reported procedure by Pourali and Ghanei [38]. All of the products were characterized by comparison of their spectral (IR, ¹H NMR and ¹³C NMR) and physical data with authentic samples.

Oxidation of Didodecylsulfane to 1-(Dodecylsulfinyl)dodecane Using PVP-H₂O₂, Silica Sulfuric Acid and Ammonium Bromide

PVP-H₂O₂ (0.290 g), silica sulfuric acid (0.100 g), ammonium bromide (0.050 g, 0.05 mmol) and water (2 drops) were added to a solution of didodecylsulfane (0.371 g, 1 mmol) in acetonitrile (10 ml). The resulting mixture was stirred at room temperature for 90 min (the reaction progress was monitored by TLC) and then filtered. The residue was washed with CH₂Cl₂ (20 ml). The reaction mixture was dried, washed with water (5 × 10 ml), then resolved in dichloromethane. Anhydrous Na₂SO₄ (3 g) was added to the filtrate and filtered out after 20 min. Finally organic solvents were evaporated and 1-(dodecylsulfinyl)dodecane was obtained as a white crystalline solid (0.383 g, 99%); m.p.: 89-91 °C; ¹H NMR (500 MHz, CDCl₃): 2.70-2.57 (m, 4H), 1.70-1.80 (m, 4H), 1.49-1.40 (m, 4H), 1.39-1.21 (m, 32H), 0.89-0.86 (t, J = 11.5 Hz, 6H) ppm; ¹³C NMR (125 MHz, CDCl₃): 52.5, 31.9, 29.6, 29.5, 29.3, 29.3, 29.2, 28.9, 22.6, 22.6, 22.5, 14.1 ppm.

Oxidation of 4-Bromobenzyl Alcohol into 4-Bromobenzaldehyde Using PVP-H₂O₂, Silica Sulfuric Acid and Ammonium Bromide

PVP-H₂O₂ (0.290 g), silica sulfuric acid (0.100 g), ammonium bromide (0.050 g, 0.05 mmol) and water (2 drops) were added to a solution of 4-bromobenzyl alcohol (0.187 g, 1

mmol) in acetonitrile (10 ml). The resulting mixture was stirred at 60 °C for 90 min (the reaction progress was monitored by TLC). Then the crude product was purified by short column chromatography using dichloromethane as the eluent. Finally, CH₂Cl₂ was evaporated and 4-bromobenzaldehyde was obtained in 88% yield (0.163 g). ¹H NMR (400 MHz, CDCl₃): δ = 9.98 (s, 1H), 7.76-7.74 (d, J = 8.4 Hz, 2H), 7.70-7.68 (d, J = 8.4 Hz, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ = 191.0, 135.1, 132.4, 130.9, 129.7 ppm.

RESULTS AND DISCUSSION

In order to complete our studies on the application of polymeric-supported reagents [39-42] in organic functional group transformations, we attempted to investigate the chemoselective oxidation of sulfides and alcohols into sulfoxides and carbonyl compounds by polyvinylpyrrolidone-supported hydrogen peroxide (PVP-H₂O₂). PVP-H₂O₂ was prepared *via* the reported procedure by Pourali and Ghanei [38].

Our recent studies are aimed at *in situ* generation of bromonium ion (Br⁺) and its applications in different organic reactions [43-47]. To this end, we decided to design a new metal-free catalytic system including polyvinylpyrrolidone-supported hydrogen peroxide (PVP-H₂O₂), silica sulfuric acid and ammonium bromide as the catalyst for the *in situ* generation of Br⁺. To examine the oxidizing properties of this catalytic media, we applied this system to the oxidation of alcohols and sulfides.

Initially, in order to find the appropriate solvent for the oxidation of alcohols and sulfides, we designed separated reactions for these transformations in different organic solvents. Dibenzyl sulfide and 4-bromobenzyl alcohol were subjected to the oxidation reaction by PVP-H₂O₂, silica sulfuric acid and a catalytic amount of ammonium bromide in the presence of two drops of water. The results of these reactions are summarized in Table 1.

From the solvent screening (Table 1) acetonitrile emerged as the most convenient one due to the fact that the products were isolated in almost quantitative yields and shortest reaction times. Therefore, acetonitrile was selected as the reaction solvent for all of the oxidation reactions. Also, as is

Table 1. Oxidation of Dibenzyl Sulfide and 4-Bromobenzyl Alcohol by PVP-H₂O₂, Silica Sulfuric Acid and Ammonium Bromide in the Presence of Two Drops of Water in Different Solvents^a

Entry	Compound	Solvent/Temperature	Time (min)	Yield (%) ^b
1	Dibenzyl sulfide	Acetonitrile/r.t.	90	98
2	Dibenzyl sulfide	Acetone/r.t.	24 h	trace
3	Dibenzyl sulfide	Chloroform/r.t.	24 h	- ^c
4	Dibenzyl sulfide	Dichloromethane/r.t.	24 h	- ^d
5	Dibenzyl sulfide	<i>n</i> -Hexane/r.t.	24 h	- ^e
6	Dibenzyl sulfide	Ethyl acetate/r.t.	25 h	73 ^f
7	Dibenzyl sulfide	Ethanol/r.t.	6 h	67
8	4-Bromobenzyl alcohol	Ethanol/60 °C	180	Sluggish
9	4-Bromobenzyl alcohol	Acetone/60 °C	195	85
10	4-Bromobenzyl alcohol	Chloroform/60 °C	90	Sluggish
11	4-Bromobenzyl alcohol	<i>n</i> -Hexane/60 °C	80	36
12	4-Bromobenzyl alcohol	Ethyl acetate/60 °C	240	51
13	4-Bromobenzyl alcohol	Acetonitrile/60 °C	90	88
14	4-Bromobenzyl alcohol	Acetonitrile/40 °C	180	70
15	4-Bromobenzyl alcohol	Acetonitrile/rt	200	73

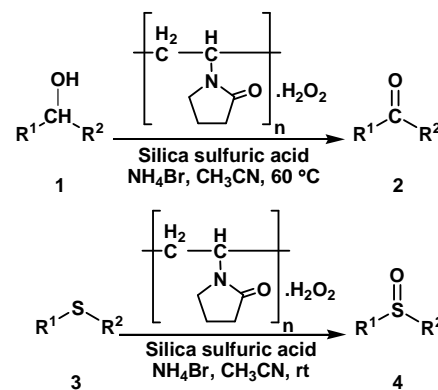
^aSubstrate/PVP-H₂O₂/silica sulfuric acid/ammonium bromide: 1 mmol/0.290 g/0.1 g/0.05 mmol).

^bIsolated yield. ^cReaction didn't complete. ^dReaction didn't complete and impurity of sulfone was observed on TLC. ^eNo reaction. ^fGC yield.

evident from Table 1, the highest yield for the oxidation of 4-bromobenzyl alcohol was obtained at 60 °C; thus this temperature was used in all alcohol oxidation reactions.

With the optimal conditions in hand, a wide variety of sulfides **1** and alcohols **3** were selectively oxidized into their corresponding sulfoxides **2** and aldehydes or ketones **4** using polyvinylpolypyrrolidone-supported hydrogen peroxide (PVP-H₂O₂), silica sulfuric acid and catalytic amounts of ammonium bromide at appropriate temperature (Scheme 1 and Table 2).

Sulfoxidation and oxidation of alcohols heterogeneously proceeded under mild conditions. Oxidation of sulfides was readily carried out *via* mixing of a sulfide with PVP-H₂O₂, silica sulfuric acid, catalytic amounts of ammonium bromide and two drops of water and stirring of this mixture at room temperature for the appropriate time. The pure product was obtained easily by filtration and evaporation of the organic solvent. On the other hand, the oxidation of alcohols was performed by the same mixture at 60 °C. After the completion of the reaction, the corresponding aldehyde or ketone was

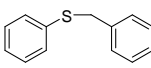
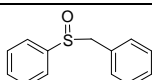
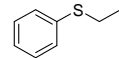
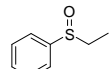
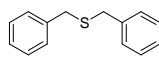
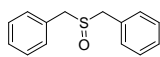
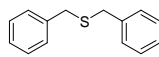
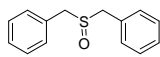
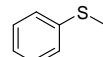
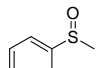
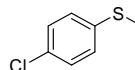
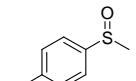
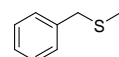
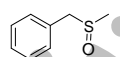
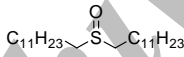


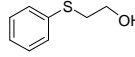
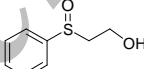
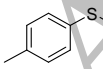
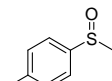


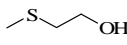
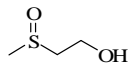


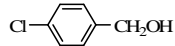
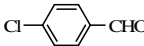


Scheme 1. Catalytic oxidation of alcohols and sulfides

easily obtained by passing the reaction mixture through a short column.

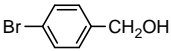
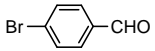
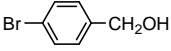
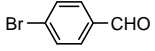
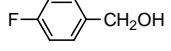
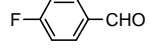
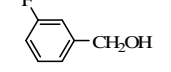
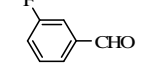
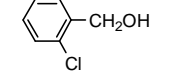
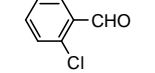
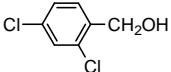
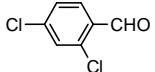
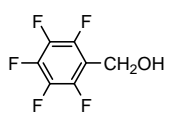
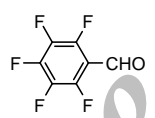
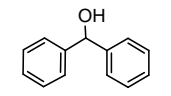
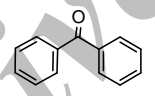
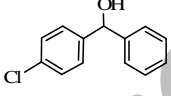
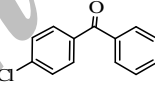
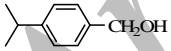
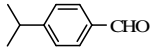
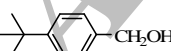
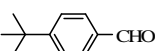
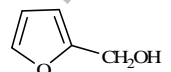
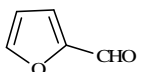
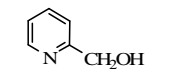
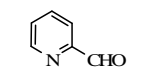
In order to prove the catalytic role of ammonium bromide in the described oxidizing systems, dibenzyl sulfide and 4-bromobenzyl alcohol (as typical examples) were subjected to

Table 2. Oxidation of Sulfides and Alcohols into Sulfoxides and Carbonyl Compounds, Respectively, Using PVP-H₂O₂, Silica Sulfuric Acid and Catalytic Amounts of NH₄Br in the Presence of Two Drops of Water

Entry	Substrate	Product	Substrate/Reagents/Catalyst ^b			Time (min)	Yield (%) ^c
			I	II	III		
1			0.44	0.2	0.15	170	98
2			0.29	0.1	0.05	110	91
3			0.29	0.1	0.05	90	98
4			0.29	0.1	-	48 h	95 ^d
5			0.29	0.1	0.05	90	99
6			0.29	0.1	0.05	75	97
7			0.29	0.1	0.05	45	96
8	$C_{11}H_{23}-S-C_{11}H_{23}$		0.29	0.1	0.05	90	99
9			0.29	0.1	0.05	30	83
10			0.29	0.1	0.05	160	93
11			0.29	0.1	0.05	105	98
12			0.29	0.1	0.05	20	89
13			0.29	0.1	0.05	70	95
14			0.29	0.1	0.05	45	99
15			0.29	0.1	0.05	120	81

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Table 2. Continued

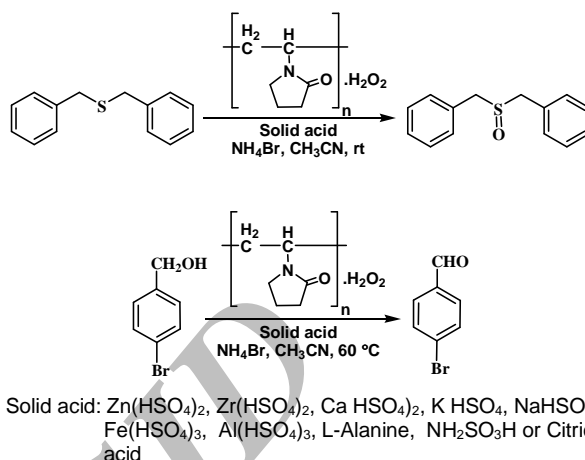
16			0.29	0.1	0.05	90	88
17			0.29	0.1	-	300	Trace ^d
18			0.29	0.1	0.05	180	93
19			0.29	0.1	0.05	180	90
20			0.29	0.1	0.05	120	80
21			0.29	0.1	0.05	120	94
22			0.29	0.1	0.05	180	Trace
23			0.29	0.1	0.05	140	94
24			0.29	0.1	0.05	70	95
25			0.29	0.1	0.05	120	88
26			0.29	0.1	0.05	90	86
27			0.29	0.1	0.05	120	45
28			0.29	0.1	0.05	270	N.R.

^aOxidation of alcohols proceeded at 60 °C and oxidation of sulfides carried out at room temperature. ^b**I** and **II** refer to grams of PVP-H₂O₂ and silica sulfuric acid, respectively; **III** refer to mmol of NH₄Br. ^cIsolated yield. ^dReaction proceeded in the absence of catalyst.

the oxidation reaction in the absence of the catalyst. As is evident from Table 2 (Entry 18), trace conversion of 4-bromobenzaldehyde was observed after 5 h. Also reaction time for the oxidation of dibenzyl sulfide increased from 1.5 hours to 48 h (Table 2 and entry 4).

To investigate and develop the scope and limitation of the oxidizing media, we decided to examine different solid acids, instead of silica sulfuric acid, in the described oxidizing system (Scheme 2 and Table 3).

As is evident from Table 3, a variety of solid acids bring about this transformation as silica sulfuric acid does in this oxidizing system. In order to investigate the role of solid acids in the described systems, dibenzyl sulfide and 4-bromobenzyl alcohol were subjected to the oxidation in the absence of any



Scheme 2. Oxidation of dibenzyl sulphide and 4-bromobenzyl alcohol in the presence of different solid acids

Table 3. Oxidation of Dibenzyl Sulfide and 4-Bromobenzyl Alcohol with PVP-H₂O₂, Solid Acid, Two Drops of Water and Catalytic Amounts of NH₄Br^a

Entry	Compound	Solid acid	Time (min)	Yield (%) ^b
1	Dibenzyl sulfide	Zn(HSO ₄) ₂	22 h	88
2	Dibenzyl sulfide	Zr(HSO ₄) ₄	50	99
3	Dibenzyl sulfide	Ca(HSO ₄) ₂	65	98
4	Dibenzyl sulfide	KHSO ₄	130	98
5	Dibenzyl sulfide	NaHSO ₄	230	99
6	Dibenzyl sulfide	Fe(HSO ₄) ₃	200	98
7	Dibenzyl sulfide	Al(HSO ₄) ₃	55	98
8	Dibenzyl sulfide	L-Alanine	24 h	N.R.
9	Dibenzyl sulfide	NH ₂ SO ₃ H	24 h	Trace ^c
10	Dibenzyl sulfide	Citric acid	48 h	98
11	Dibenzyl sulfide	-	240	N.R.
12	4-Bromobenzyl alcohol	Zn(HSO ₄) ₂	140	61
13	4-Bromobenzyl alcohol	Zr(HSO ₄) ₄	100	Sluggish
14	4-Bromobenzyl alcohol	Ca(HSO ₄) ₂	70	83
15	4-Bromobenzyl alcohol	KHSO ₄	200	77
16	4-Bromobenzyl alcohol	NaHSO ₄	300	67
17	4-Bromobenzyl alcohol	Fe(HSO ₄) ₃	120	82
18	4-Bromobenzyl alcohol	Al(HSO ₄) ₃	140	85
19	4-Bromobenzyl alcohol	L-Alanine	300	Trace ^c
20	4-Bromobenzyl alcohol	NH ₂ SO ₃ H	300	76
21	4-Bromobenzyl alcohol	Citric acid	300	74
22	4-Bromobenzyl alcohol	-	240	N.R.

^aSubstrate:PVP-H₂O₂:acid:NH₄Br = 1 mmol:0.290 g:0.5 mmol:0.05 mmol. ^bIsolated yield. ^cIn the absence of acid.

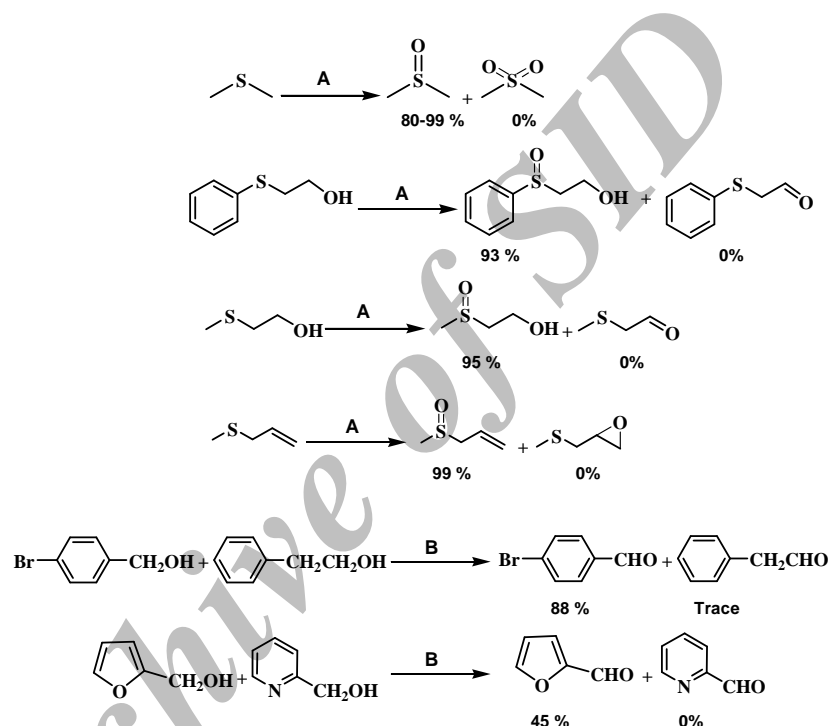
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acid; surprisingly it was observed that no reactions occurred after 4 h (Table 3, entries 11 and 22), which means that the presence of acid is necessary for these transformations.

One of the outstanding advantages of these oxidizing systems is the selectivity and chemoselectivity in several cases, which is outlined in Scheme 3. The suggested

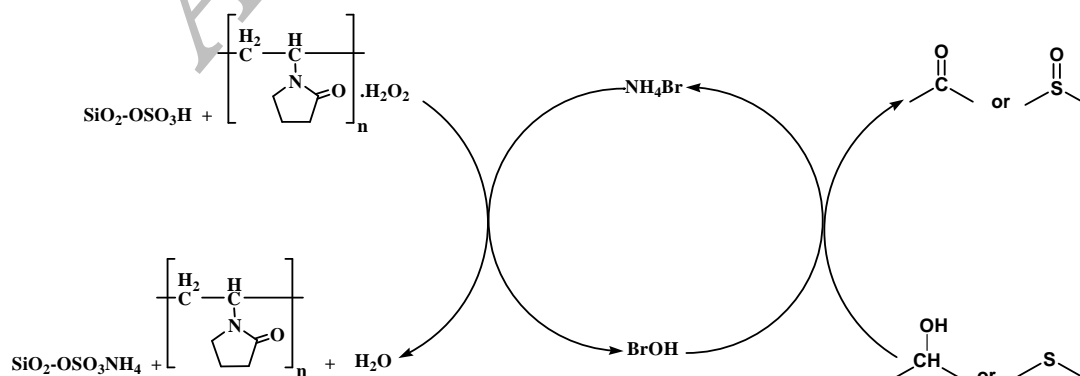
mechanism for these transformations has been outlined in Scheme 4 based on the previously reported works [43-47].

Initially, the combination of polyvinylpolypyrrolidone-supported hydrogen peroxide (PVP-H₂O₂) and silica sulphuric acid react with ammonium bromide to produce hypobromous acid (BrOH). Finally, hypobromous acid oxidizes alcohol or



A= PVP-H₂O₂ (0.29 g), Silica sulfuric acid (0.1 g), NH₄Br (0.05 mmol), H₂O (2 drops), CH₃CN, r.t.
B= PVP-H₂O₂ (0.29 g), Silica sulfuric acid (0.1 g), NH₄Br (0.05 mmol), H₂O (2 drops), CH₃CN, 60 °C

Scheme 3. Selectivity in the oxidation of alcohols and sulfides



Scheme 4. Mechanism of the oxidation reaction of alcohols and sulfides

sulfide in the presence of water.

In conclusion, the chemistry of polyvinylpyrrolidone-supported hydrogen peroxide (PVP-H₂O₂) has opened up a new methodological option for the catalytic, versatile and efficient oxidation of various types of sulfides and alcohols. Characteristics such as short reaction time, metal-free content, good reaction yields, no environmental pollution, and simple workup procedure make this method an excellent alternative to the oxidation of the sulfides into sulfoxides.

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