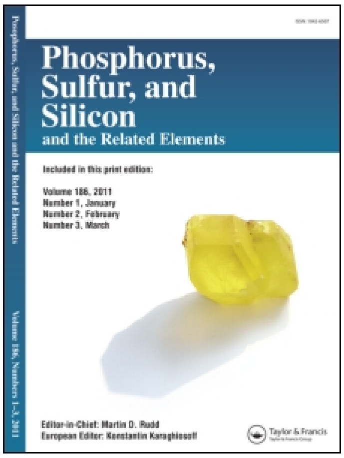

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POLYVINYLPYRROLIDONE-SUPPORTED HYDROPEROXIDE FOR SELECTIVE OXIDATION OF ALDEHYDES TO CARBOXYLIC ACIDS AND SULFIDES TO SULFOXIDES

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The facile immobilization of hydroperoxide on the cross-linked poly(vinylpyrrolidone) is described by treatment of a poly(vinylpyrrolidone)-Vilsmeier adduct with (35%) hydrogen peroxide. The in situ–generated poly(vinyl pyrrolidone)-supported hydroperoxide reagent showed very good performance in chemoselective oxidation of aldehydes to carboxylic acids as well as sulfides to sulfoxides.

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Keywords Carboxylic acid; hydrogen peroxide; poly(vinylpyrrolidone); sulfoxide; Vilsmeier reagent

INTRODUCTION

The Vilsmeier reaction has been used for the synthesis of a variety of cyanines via formylation of Fisher's bases followed by Knoevenagel condensation reaction.[1,2] Since the first report appeared in 1927, the Vilsmeier–Haack reaction has been utilized for an immense variety of activated aromatic compounds to complex heterocycles.[3–7] Recently, *t*-butyldimethylsilylated alcohols were converted in one step to the corresponding formates under Vilsmeier–Haack conditions.[8] Traditionally, Vilsmeier reagents have been utilized as a formylating agent, but in recent years their versatility has also been demonstrated as an activating agent for halogenation of β-diketones[9] and ring opening of substituted 2,3-epoxy-1,3-diphenyl propan-1-one.[10]

The ability of the Vilsmeier reagent to give rise to products of substitution with substrates bearing a labile hydrogen atom has provided some interesting applications for the preparation of alkylhalide,[11] esters,[12] acid chloride,[13] and nitriles.[14]

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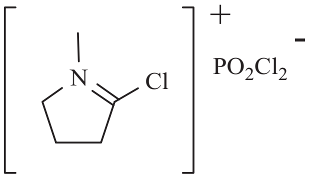
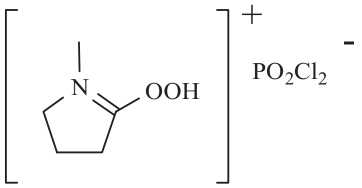
Treatment of a monomeric Vilsmeier reagent with hydrogen peroxide affords a highly electrophilic hydroperoxy methylenedimethyl-ammonium salt, which could efficiently produce 1,2-dichloro compounds of corresponding alkenes or act as a powerful epoxidizing reagent, depending on the reaction conditions.[15] A Vilsmeier reagent of *N*-methylpyrrolidone (I) prepared by reaction of NMP with $POCl_3$ affords an active oxygen transfer reagent (II) by reaction with hydrogen peroxide, which epoxidizes the double bonds of alkenes exclusively.[16] However, this reagent is a highly reactive salt, and attempts to isolate it were unsuccessful.



(I)

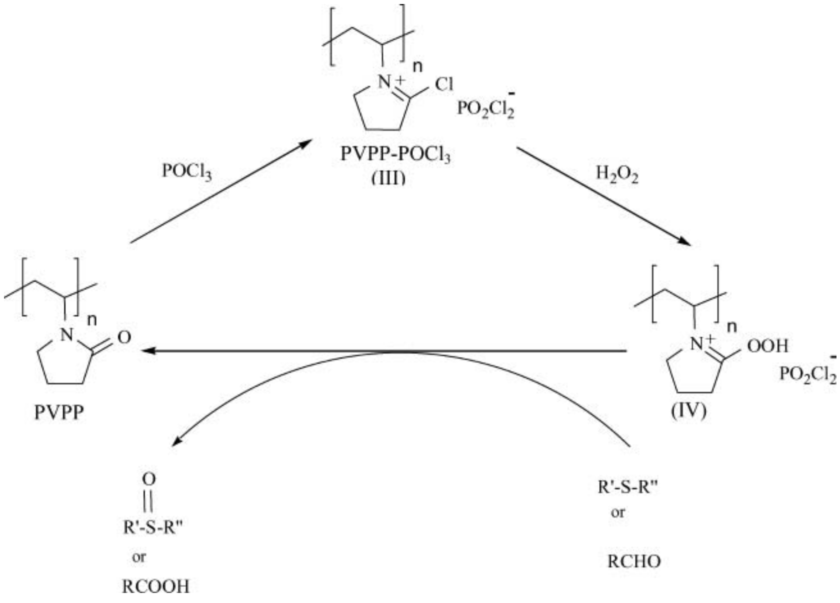


(II)

Polymer-supported catalysts and reagents have become popular in organic synthesis over the past few decades.[17] Their high catalytic activity, low toxicity, stability, recyclability, and environmentally safe conditions make the use of polymer-supported reagents attractive alternatives to conventional reagents. Hydrogen peroxide has been used for the oxidation of aldehydes to their corresponding carboxylic acids under basic conditions, alone or in combination with other reagents.[18–21] Indeed, several methods have been reported for the transformation of aldehydes to carboxylic acids.[22–27] However, these methods are all deficient in some respect, such as strong acidic or basic conditions, use of heavy metal as a catalyst, low yield, or high cost. Furthermore, most of them suffer from low selectivity. On the other hand, there are several methods for the oxidation of alcohols to aldehydes using polymer-supported reagents, whereas their further oxidation to corresponding acids has not been well demonstrated. Although the wide variety of polymer-supported catalysts and reagents have been well established for organic transformation, to the best of our knowledge there is no literature report on the use of a H_2O_2 adduct of a polymer-supported Vilsmeier-type reagent. Therefore, in continuation of our studies on the development of novel synthetic methodologies using polymeric systems and of oxidative transformations,[28–30] in this article we introduce the use of a polymeric Vilsmeier reagent (III) as an environmentally benign promoter for the oxidation of aldehydes and sulfides using hydrogen peroxide (Scheme 1).

RESULTS AND DISCUSSION

The poly(vinylpyrrolidone)-supported Vilsmeier reagent (PVPP-$POCl_3$) is obtained as a white powder by treatment of a suspension of cross-linked polyvinylpyrrolidone(PVPP) with $POCl_3$ in dichloromethane at room temperature. In contrast to the monomeric Vilsmeier reagents, which are extremely hygroscopic, this reagent is nonhygroscopic powder. However, the use of linear poly(vinylpyrrolidone) was not satisfactory for the reaction with $POCl_3$ due to the formation of a sticky resin. Characterization of the chloropyrrolidinum dichlorophosphate groups on the cross-linked reagent was performed by recording the Fourier transform infrared spectroscopy (FTIR) spectra (Supplemental Materials,



Scheme 1

Figure S1, available online). The IR spectrum shows a broad absorption at 1250–1293 cm^{-1} for the P=O bond and 750 cm^{-1} for P—Cl, 1027 and 532 cm^{-1} due to the C—Cl bond, and absorption at 1643 cm^{-1} corresponds to the C=N of iminium in the ring. Indeed, the ^{31}P NMR spectrum of the linear polyvinylpyrrolidone-supported reagent clearly indicated the characteristic δ_{p} of a dichlorophosphate anion[31] by a signal at −13.6ppm in DMSO.

To show the role of the Vilsmeier adduct in these oxidation reactions, the oxidation of *p*-methoxybenzaldehyde was carried out in the absence of the Vilsmeier reagent. The oxidation was very slow, and the *p*-methoxybenzoic acid was obtained in very poor yield. Also, the oxidation of sulfide, e.g., methylphenylsulfide, without using the Vilsmeier reagent was not successful, and a low yield of corresponding sulfoxide was formed. As seen in Table I, the optimized stoichiometric ratio of RCHO:H_2O_2 for the conversion of aldehydes to carboxylic acids was found to be 1:3, using 0.5 g of the reagent in acetonitrile at 45°C. In the oxidation of methylphenylsulfides, a ratio of 1:2 of sulfide to hydrogen peroxide in the presence of 0.25 g of the reagent produces the corresponding sulfoxide in excellent yield in methanol at room temperature.

To select the best solvent, the oxidation of the *p*-methoxybenzaldehyde and methylphenylsulfide were studied in a variety of organic solvents such as methanol, acetonitrile, dioxane, and dichloromethane. Among them, acetonitrile and methanol were found to be the best solvents for oxidation of aldehydes and sulfides, respectively (Table II). The oxidation reaction of aldehydes was found to be slow at room temperature, and very low yield of product was obtained, even at longer reaction times. A protic solvent such as methanol is required to facilitate the oxygenation of sulfide to give the corresponding sulfoxide in excellent yield.

To explore the oxidation ability of the in situ–generated, polymer-supported hydroperoxide reagent (IV), the oxidation of a variety of aromatic and aliphatic aldehydes

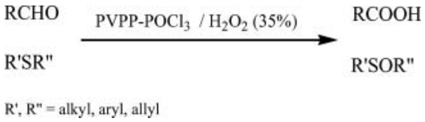
Table I Effect of PVPP-$POCl_3$ on oxidation of *p*-methoxybenzaldehyde and methylphenyl sulfide[a]

Entry	Substrate (1 mmol)	H_2O_2 (mmol)	PVPP-$POCl_3$ (g)	Yield (%)
1	*p*-MeO-C_6H_4CHO[a]	1	0.5	38
2	”	2	0.5	62
3	”	3	0.0	15
4	”	3	0.25	26
5	”	3	0.35	38
6	”	**3**	**0.5**	**82**
7	”	4	0.5	88
8	Ph-S-Me[b]	1	0.25	96
9	”	**2**	**0.25**	**98**
10	”	3	0.25	98
11	”	2	0.0	20
12	”	2	0.13	90
13	”	2	0.2	95
14	”	2	0.25	96

[a] Reactions were performed three runs in acetonitrile at 45°C in 12 h.

[b] Reactions were performed three runs in methanol at room temperature in 10 min.

was examined by treating the substrate with polyvinylpyrrolidone-supported Vilsmeier reagent in the presence of 35% hydrogen peroxide in acetonitrile at 45°C (Scheme 2).



Scheme 2

These results are presented in Table III. As is evident, all the aliphatic aldehydes were selectively and easily converted to the corresponding acids in excellent yields. Aromatic aldehydes were converted to the corresponding acids in moderate to good yields at longer periods. The presence of other functional groups such as hydroxyl and double bond

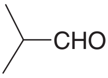
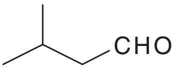
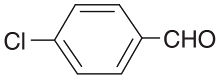
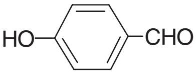
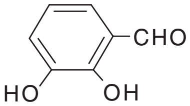
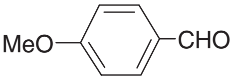
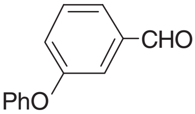
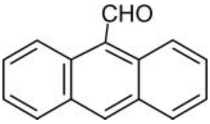
Table II Oxidation of methyl phenyl sulfide and *p*-methoxybenzaldehyde in different solvents

Entry	Substrate (1 mmol)	Solvent	Reaction time	Yield (%)
1	PhSMe[a]	**CH_3OH**	**10 min**	**98**
2	”	CH_3CN	25 min	92
3	”	Dioxane	45 min	75
4	”	CH_2Cl_2	25 min	65
5	*p*-MeOC_6H_4-CHO[b]	CH_3OH	12 h	65
6	”	**CH_3CN**	**12 h**	**75**
7	”	Dioxane	12 h	68
8	”	CH_2Cl_2	12 h	55

[a] Reactions were carried out using 0.25 g of the polymeric reagent and 2 mmol of H_2O_2.

[b] Reactions were carried out using 0.5 g of the polymeric reagent and 3 mmol of H_2O_2.

Table III Oxidation of aldehydes to carboxylic acids using (PVPP-$POCl_3$)/H_2O_2[a]

Entry	Aldehyde	Time (h)	Yield%[b]	Ref.
1	$CH_3CH_2CH_2CH_2$ CH_2CHO	1	98	32,33
2		1	98	32,33
3		1	98	32
4	Ph-CHO	12	75	32,33
5		12	72	32,33
6	PhCH = CH−CHO	12	85	33
7	PhCH = C(Me)-CHO	12	87	33
8		12	78	33
9		12	82	33
10		12	82	32,33
11		1	76	33
12		1	78	33

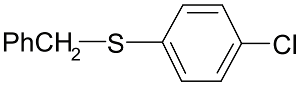
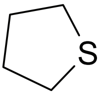
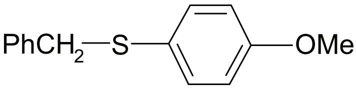
[a] Reaction conditions: substrate (1 mmol), PVPP-$POCl_3$ (0.5 g), H_2O_2 (3 mmol), MeCN (45°C).
[b] Isolated yields.

(Table III, entries 6–9) was found to be inert, and aldehydes were selectively oxidized to the acids without affecting these groups.

Finally, we examined the oxidation reaction of structurally different sulfides with a Vilsmeier reagent/H_2O_2 system in methanol at room temperature (Scheme 2). All reactions were carried out under the optimized conditions, and the results are summarized in Table IV. Reaction times are very short, and the sulfoxides are obtained in excellent yields. It is clear that sulfides bearing sensitive functional groups such as hydroxy, methoxy, carbaldehyde, and carbon–carbon double bonds are completely unaffected under the reaction conditions.

As shown in Scheme 1, after completion of the reactions and workup, the combined spent resin was filtered off, and the phosphate species are removed from the reagent. Upon

Table IV Oxidation of sulfides to sulfoxides using (PVPP-$POCl_3$)/H_2O_2[a]

Entry	Sulfide	Time (min)	Yield%[b]	Ref.
1	Ph-S-Me	10	99	34
2	Ph-CH_2-S-CH_2-Ph	15	98	34
3	PhCH_2-S-Ph	25	97	35
4	Ph-S-Ph	40	94	32,33
5		35	92	33
6	Ph-S-CH_2CH_2OH	25	95	36,47
7	Me-S-CH_2CH_2-CHO	5	98	47
8	$(CH_3CH_2CH_2)_2S$	5	99	37
9	$(CH_3CH_2CH_2CH_2)_2S$	5	99	38
10	$(CH_2=CH{-}CH_2)_2S$	5	98	39,47
11		5	98	40
12		20	95	29

[a]Reaction conditions: sulfide (1 mmol), PVPP-$POCl_3$ (0.25 g), H_2O_2 (2 mmol), MeOH, r.t.
[b]Isolated yields.

treatment of the recycled PVPP with a fresh $POCl_3$ solution, the Vilsmeier reagent was regenerated and reused for the new oxidation reactions.

Investigation about the use of the polyvinylpyrrolidone-supported Vilsmeier reagent and hydrogen peroxide for oxygenation of alkenes to corresponding epoxides is underway.

In order to show the advantages and disadvantages of our method, some of our results in oxidation of sulfides and aldehydes were compared with some of those reported in literature[26–41] (Table V). It is evident that the PVPP-$POCl_3$/H_2O_2 system allows these transformations to proceed with excellent selectivity, good yield, shorter times, and milder conditions. Furthermore, the recyclability of the polymer support together with its biocompatibility makes the method environmentally safe and friendly.

In summary, we demonstrated poly(vinylpyrrolidone)-phosphorous oxychloride as a versatile polymeric Vilsmeier reagent that exhibits excellent selectivity toward oxygenation of sulfides to sulfoxides, and oxidation of aldehydes to carboxylic acids in the presence of hydrogen peroxide under mild reaction conditions. This polymeric Vilsmeier reagent was found to retain its activity after months and is stable in a glass bottle at room temperature. In addition, this method is suitable both for preparative and industrial usage because of its low cost, biocompatibility, and recyclability of the poly(vinylpyrrolidone) as a safe polymer support.

EXPERIMENTAL

Melting points were recorded on an electrothermal melting point apparatus and are uncorrected. The NMR spectra were recorded in $CDCl_3$ with TMS as an internal standard

Table V Comparison of PVPP-$POCl_3$/H_2O_2 with some other reagents for oxidation of sulfides and aldehydes to corresponding sulfoxide and carboxylic acids, respectively

Substrate	Reagent	Conditions	Time	Yield (%)[a]	Ref.
Ph-S-Me	H_2O_2(35%), $TiCl_3$	H_2O/MeOH, r.t.	5 min	100	41
”	Caro's acid/SiO_2	MeCN, reflux	4.5 h	92	42
”	Oxone (3 eq.)	H_2O/MeOH, 0°C	4 h	95[a]	43
”	$KMnO_4/MnO_2$	CH_2Cl_2, r.t.	3 h	93[a]	44
”	$NaIO_4$	H_2O/0°C	15 h	91	45
”	t-BuOOH, Ti(O-Pr)$_4$	CH_2Cl_2, −20°C	5 h	80	46
”	**PVP-$POCl_3$/H_2O_2**	**MeOH, r.t.**	**10 min**	**98**	
”	NAHBP[b]	THF/ H_2O, r.t.	1 h	91	47
PhCHO	Oxone/H_2SO_4	$H_20/CHCl_3$, r.t.	48 h	70	48
”	MnO_2	Petroleum ether/reflux	24 h	75	49
”	$PhCH_2NEt_3MnO_4$	AcOH, r.t.	1.25 h	84	50
”	NiO_2/NaOH	H_2O, 30°C	1.5 h	94	51
”	UHP (4eq)[c]	HCOOH(95%)/r.t.	1.5 h	96	22
”	Ni(dmp)$_2$/O_2 (1atm)	Acetone, 0°C	6 h	78.6	52
”	PB-SiO_2/$KMnO_4$	Cyclohexane, 65°C	20 h	99	53
”	H_2O_2/$MeReO_3$	[bmim]BF_4, 50°C	24 h	95	41
	MPHT[d] (10%)/H_2O_2	MeCN, 80°C	1.5 h	98	54
”	**PVP-$POCl_3$/H_2O_2**	**MeCN, 45°C**	**1 h**	**97**	

[a] Yield refers to the corresponding sulfone.

[b] NAHBP = nicotinic acid hydrobromide perbromide.

[c] UHP = urea hydrogen peroxide.

[d] MPHT = methylpyridin-2-one hydrobromide.

on Bruker WM 250 and 400 MHz NMR spectrometers. IR spectra were determined on a SP-1100, P-UV-Com instrument. 20% cross-linked polyvinylpyrrolidone (PVPP) and linear PVPP ($M_w = 90000$) were purchased from Fluka. Elemental analyses were performed by a CHN-O rapid Heraeus elemental analyzer (Wellesley, MA). Purity determination of the products was accomplished by TLC on silica gel polygram SIL G/UV 254 plates. Products were identified by the comparison of their mp, IR, and NMR spectra with those reported for authentic samples.

Preparation of Polyvinylpyrrolidone-Supported Phosphorus Oxychloride (PVPP-$POCl_3$)

To a suspension of cross-linked polyvinylpyrrolidone (PVPP, 4g) in CH_2Cl_2 (25 mL), a solution of phosphorus oxychloride (5 mL) in CH_2Cl_2 (15 mL) was added dropwise, and the mixture was stirred for 0.5 h at room temperature. The resulting resin was filtered and washed with CH_2Cl_2 (2 × 10 mL), and was finally dried in desiccator to give stable and non-hygroscopic powder. CHN: Anal. Calcd. C, 27.22; N, 5.65; H, 3.38. Found: C, 2734; N, 5.41; H, 3.58. IR (KBr): 3399, 2952, 1644, 1543, 1516, 1474, 1293, 1224, 1027,750, 535cm^{-1}. ^{13}C NMR, (100.6 MHz, $CDCl_3$): $\delta = 176$ (-(Cl)C=N$^+$-). ^{31}P NMR (161.8MHz, DMSO-d$_6$): $\delta = -13.6$ ($PO_2Cl_2^-$). Capacity of the reagent was determined by iodometric titration and was found to be 3.7 mmol of $PO_2Cl_2^-$ per gram of polymeric reagent.

General Procedure for Oxidation of Aldehydes to Carboxylic Acids

To a suspension of aldehyde (1 mmol) and PVPP-$POCl_3$ (0.5 g) in acetonitrile (5 mL), 35% hydrogen peroxide (3 mmol) was added dropwise. The reaction mixture stirred for 1–12 h at 45°C. After completion of the reaction (TLC), the mixture was filtered and purified by silica gel column chromatography to afford the desired products in 72–98% yields.

General Procedure for Oxidation of Sulfides

To a suspension of sulfide (1 mmol) and PVPP-$POCl_3$ (0.25 g) in methanol (5 mL), 35% hydrogen peroxide (2 mmol) was added dropwise. The reaction mixture was stirred for 10–45 min at room temperature. After completion of the reaction (TLC), the mixture was filtered and washed with H_2O (5 mL). The filtrates were extracted with diethyl ether (20 mL), and the combined organic fraction was washed with 3% $NaHSO_3$ and dried over Na_2SO_4. Evaporation of the solvent gave the corresponding sulfoxide in 92–99% yields.

Benzyl (4-Methoxyphenyl) Sulfoxide

Mp = 105–107°C. IR(KBr)(ν_{max}, cm^{-1}): 3090, 2992, 2832, 1442, 1305, 1147, 1037, 750, 660. ^1H NMR (400MHz, DMSO-d_6): $\delta = 7.8$(d, 2H), 7.75(d, 2H), 7.47(m, 3H), 7.3(m, 2H), 4.0(s, 3H), 3.8(d, 1H), 4.2(d, 1H).

Regeneration of Polyvinylpyrrolidone-Supported Phosphorus Oxychloride (PVPP-$POCl_3$)

The spent reagent collected from each oxidation reaction was washed thoroughly with water and diethyl ether to remove any residual compounds. The resin was filtered and dried in vaccum at 50°C. The recycled polyvinylpyrrolidone was treated with $POCl_3$ in dichloromethane and was stirred for 0.5 h at room temperature. Then, the mixture was filtered and washed with dichloromethane and dried in a desiccator.

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