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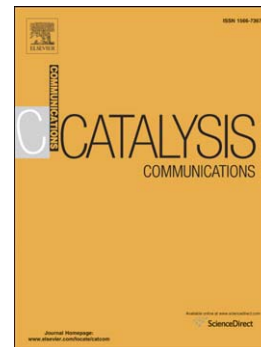
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# Highly Efficient and Selective Aerobic Oxidation of Alcohols in Aqueous Media by TEMPO-Containing Catalytic Systems

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## Abstract

The use of molecular oxygen as the oxidant and water as reaction medium is a charming research orientation in the oxidation of alcohols. In this communication, the efficient aerobic oxidation of alcohols to the corresponding carbonyl compounds in aqueous media has been achieved with TEMPO-containing catalytic systems. In absence of base/acid, a 99% conversion and 94% selectivity for benzaldehyde was obtained for the oxidation of benzyl alcohol by TEMPO-Ce(IV)-NaNO<sub>2</sub> in water. Moreover, the effects of quaternary ammonium salts and anionic surfactants were investigated. The aerobic selective oxidation of different non-heterocyclic alcohols in aqueous media was further investigated, and the moderate to high yields (45.5-98.0%) of products were obtained.

**Keywords:** Oxidation; Alcohols; TEMPO; Molecular Oxygen; Water

## 1. Introduction

The selective oxidation of alcohols to their corresponding carbonyl compounds is one of the most important transformations in industrial production and fine chemical processes [1-2]. The traditional methods involve stoichiometric oxidants such as permanganate, chromic acid and potassium dichromate, etc. [3-5]. These reagents are toxic and hazardous, and the processes will generate many inorganic or organic waste. Considering both economical and environmental benefits, the use of molecular oxygen as terminal oxidant received great attentions in recent years. Correspondingly, many catalytic systems have been developed for the aerobic alcohol oxidation [4, 6]. Among various catalytic systems, the notable heterogeneous catalysts include ruthenium catalyst [7], palladium nanoclusters [8], gold nanoparticles [9, 10], manganese oxides [11] and cobalt oxide [12], etc.. While, in homogeneous catalyst systems, the combination of 2,2,6,6-tetramethyl piperidine-1-oxyl (TEMPO) and some mediators is attractive and promising for the aerobic oxidation of alcohols [13]. However, in TEMPO-catalyzed processes, organic solvents or ionic liquids are often employed as the solvents. With toluene as the solvent, both Punniyamurthy's group and Repo's group [14, 15] found that Cu(II) complex and TEMPO was efficient for the aerobic oxidation of aromatic and allylic alcohols. With acetic acid as the solvent, Mn(II)-Co(II) nitrates and TEMPO could promote the selective oxidation of alcohols to aldehydes and ketones by oxygen [16]. In acetonitrile solvent, TEMPO and Cu(II)-triethanolamine complexes allowed efficient oxidation of primary alcohols to the corresponding aldehydes [17]. Furthermore, Stahl et al. [18] found that a (2,2-bipyridine)CuI/ TEMPO catalyst system with N-methylimidazole catalyzed the

oxidation of benzylic, allylic and aliphatic primary alcohols to aldehydes in excellent yields. In dichloromethane solvent, the TEMPO and hydroxylamine could efficiently catalyze the aerobic oxidation of alcohols [19]. Besides, in trifluorotoluene solvent, Liang and co-workers [20] found that  $\text{FeCl}_3\text{-NaNO}_2\text{-TEMPO}$  system could promote the oxidation of benzylic alcohols and cinnamyl alcohol in excellent yields. In ionic liquids, the  $\text{CuCl/TEMPO/base}$  catalytic system showed good activity for the oxidation of benzylic alcohols, and 57-99% conversion were obtained [21]. Also, the same group found that molecular sieve 3A could replace the base and promote the oxidation of alcohol in  $[\text{bmim}][\text{PF}_6]$  ionic liquid [22]. On the other hand, the two-phase reaction systems have been reported for the aerobic oxidation of alcohols. For example, in the presence of TEMPO, the fluoruous biphasic system [23, 24] and the mixture of acetonitrile and water [25, 26] were employed as the solvents for the oxidation of alcohol.

In order to avoid the safety issues associated with the flammability of mixture of oxygen and organic solvent in the gas phase [27], employing the green and sustainable water as the solvent are more promising and favorable [28]. In the former investigations, several catalytic systems have been found to be useful in water; however, the processes need to be performed in aqueous alkaline solution [29], in the presence of  $\text{AcOH}$  [30] or with hypervalent iodine as the additive [31]. Very recently, the efficient oxidation of primary benzylic alcohols in distilled water was performed in the presence of  $\text{CuBr}_2$ , *N*-isopropyl-3,5-di-*tert*-butylsalicyl aldimine (**HL3**) and TEMPO [32]. But, the preparation of **HL3** ligand is not easy.

In this communication, a new TEMPO-containing catalytic system has been developed for efficient aerobic oxidations of different alcohols in water, in which the corresponding aldehydes are obtained as main products in the presence of the TEMPO-Ce(IV)-NaNO<sub>2</sub> system under mild conditions.

## 2. Experimental

### 2.1. General reagents and equipments

Benzyl alcohol, TEMPO, benzaldehyde, Ceric ammonium nitrate (CAN) and NaNO<sub>2</sub> are of analytical grade and purchased from Alfa Aesar. The organic solvents and tetraethyl ammonium bromide (TEAB), tetraethylammonium chloride (TEAC), tetramethyl ammonium bromide (TMAB), tetramethyl ammonium chloride (TMAC), tetra butyl ammonium bromide (TBAB) , tetra butyl ammonium chloride (TBAC), dodecyl trimethyl ammonium bromide (DTAB), sodium dodecyl sulfonate (SDS), sodium dodecyl benzene sulfonate (SDBS) are of analytical grade and obtained from commercial. The deionized water is purchased from commercial and used without further treatment. Oxygen supplied in a high-pressure cylinder was used through a reducing valve without further treatment.

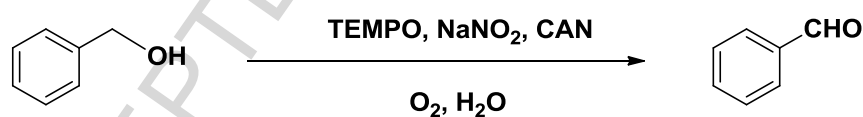
### 2.2 General Procedure for the Oxidation of Alcohols

All oxidation experiments are performed in a 120 mL autoclave equipped with the magnetic stirring and temperature control. A typical procedure for the oxidation of benzyl alcohol is as follows: 1.08 g (10.0 mmol) of benzyl alcohol, 0.0156 g (0.1 mmol) of TEMPO, 0.274 g (0.5 mmol) of CAN, 0.0690 g (1.0 mmol) of NaNO<sub>2</sub>, additive in suitable amount and 10 mL of H<sub>2</sub>O were charged into the reactor, and the

atmosphere inside is replaced with oxygen after the reactor is sealed. Then, pure oxygen is charged to 0.3 MPa at room temperature. In the following, the autoclave is heated to 80 °C under stirring, and then kept for 2 h. After reaction, the autoclave was cooled to room temperature and excess gas was purged. The mixture was transferred into a flask, in which the reactor was washed with CH<sub>2</sub>Cl<sub>2</sub> for 3-5 times in order to transfer completely. Next, the products are extracted with 6 mL CH<sub>2</sub>Cl<sub>2</sub> three times. The obtained products were analyzed with internal standard technique by GC with a flame ionization detector (All products were determined on GC-MS with Agilent 6890N GC/5973 MS detector).

### 3. Results and discussion

#### 3.1. Oxidation of benzyl alcohol with TEMPO in water



**Scheme 1.** The oxidation of benzyl alcohol with TEMPO-containing catalytic systems

**Table 1.** Oxidation of benzyl alcohol with different catalytic systems<sup>a</sup>

Entry	TEMPO	CAN	NaNO <sub>2</sub>	Conv. (%) <sup>b</sup>	Sel. (%) <sup>c</sup>
1	×	×	×	2.7	34.8
2	√	×	×	11.5	36.1
3	×	√	×	8.6	13.8
4	×	×	√	10.8	31.9
5	√	√	×	14.6	50.3
6	×	√	√	12.0	18.5
7	√	×	√	61.1	89.5
8	√	√	√	99.0	94.0

<sup>a</sup> Reaction conditions: 1.08 g benzyl alcohol in water (10 mL), in the presence of 1.0 mol% TEMPO, 5.0 mol% CAN, 10.0 mol% NaNO<sub>2</sub> and ETAB (20 mol%) under 0.3 MPa of O<sub>2</sub>,

temperature 80 °C, time 6h. The symbol “√<sup>b</sup>” represents “being added”; the symbol “×” represents “not being added” in the oxidation.

<sup>b</sup> Conv. means the conversion of benzyl alcohol. The results are obtained by GC with internal standard method.

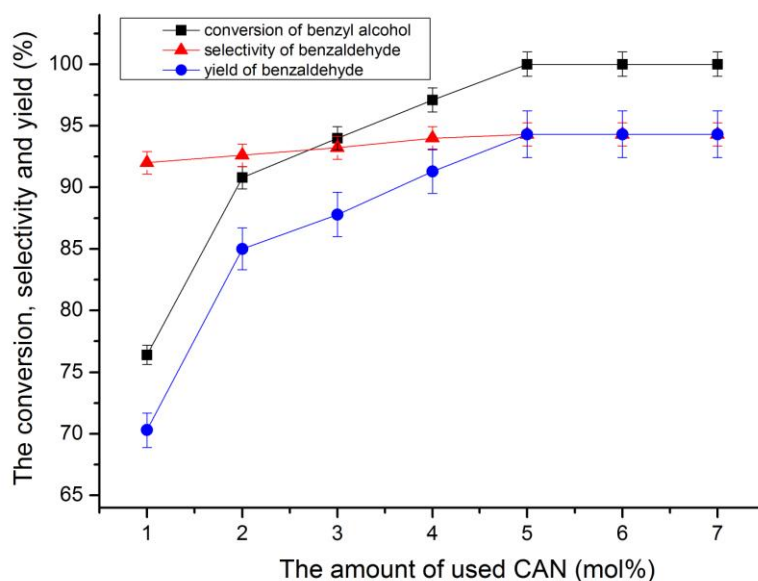
<sup>c</sup> Sel. Means the selectivity of benzaldehyde. The results are obtained by GC with internal standard technique.

In order to investigate the performance of different catalytic systems in water, the oxidation of benzyl alcohol in the presence of TEAB is selected as a model reaction (Scheme 1).

From the obtained experimental results in Table 1, the conversion of benzyl alcohol is only 2.7% in the absence of any catalyst at 80 °C for 6 h (entry 1). The conversion is elevated to 11.5% when 1.0 mol% TEMPO is added (entry 2). Besides, in the presence of single CAN or NaNO<sub>2</sub>, an 8.6% or 10.8% conversion is obtained, respectively (entries 3 and 4). In the following, the combination of two compounds is used for the oxidation of benzyl alcohol, in which the conversion of benzyl alcohol is respectively 14.6% or 12.0% when the combination of TEMPO-CAN or CAN-NaNO<sub>2</sub> is employed (entries 5 and 6). While, a 61.1% conversion in 89.5% selectivity for benzaldehyde is obtained when the TEMPO-NaNO<sub>2</sub> is used (entry 7). Furthermore, the conversion and selectivity for benzaldehyde are respectively increased to 99.0% and 94.0% when the TEMPO-CAN-NaNO<sub>2</sub> catalytic system is employed (entry 8). Moreover, no acids are detected from the obtained products by GC-MS technique. These results indicate that there exists the synergistic effect between the component TEMPO, CAN and NaNO<sub>2</sub> in water. The oxidation process is suggested as follows: the TEMPO oxoammonium salt reacts with benzyl alcohol to give the product benzaldehyde. In the following, Ce(IV) functions with TEMPOH to produce the

TEMPO oxoammonium salt and Ce(III). Then,  $\text{NO}_2$  that is originated from  $\text{NaNO}_2$  drives the oxidation of Ce(III) to Ce(IV), followed by the reaction of NO with  $\text{O}_2$  generating  $\text{NO}_2$  in which the catalytic cycle is realized.

### 3.2. Effect of the amount of CAN



**Figure 1.** Effect of the amount of CAN in the oxidation of benzyl alcohol.

Figure 1 shows the effect of different amounts of CAN in the oxidation of benzyl alcohol. Both the conversion and product yield are gradually increased along with the amount of CAN being increased from 1.0 mol% to 5.0 mol%. However, the selectivity of benzaldehyde almost kept unchanged. The maximum conversion and yield of benzaldehyde is 99.0% and 93.0% in the presence of 5.0 mol% CAN, respectively. When the amount of CAN was further increased to 7.0 mol%, the conversion and product yield are hardly changed. Thus, the suitable amount of CAN is 5.0 mol% for the oxidation of benzyl alcohol.

### 3.3. Effect of different additives



As we know, some surfactants can often accelerate the substrate to dissolve in aqueous media and promote the oxidation reaction based on the formation of the micro-reaction environment. Thus, the effects of different additives including quaternary ammonium salts and several anionic surfactants have been studied. Table 2 presents the effect of different additives in the oxidation of benzyl alcohol.

**Table 2.** Effects of different additives in the oxidation of benzyl alcohol <sup>a</sup>

Entry	Additive	Conversion (%) <sup>b</sup>	Selectivity (%)	Yield (%)	TOF (h <sup>-1</sup> ) <sup>c</sup>
1	TMAC	63.1	97.8	61.7	42
2	TMAB	99.0	99.0	98.0	66
3	TEAC	65.9	98.0	64.6	44
4	TEAB	99.0	93.7	92.8	66
5	TBAC	40.3	96.6	38.9	27
6	TBAB	99.0	92.9	92.0	66
7	DTAB	72.3	93.1	67.3	48
8	SDBS	99.0	99.0	98.0	66
9	SDS	55.3	87.6	48.4	37

<sup>a</sup> Reaction conditions: 1.08 g benzyl alcohol, in 10 mL water, 1.0 mol% TEMPO, 5.0 mol% CAN and 10.0 mol% NaNO<sub>2</sub>, in the presence of 1.0 mol% additive, under 0.3 MPa of O<sub>2</sub>, temperature 80 °C, time 1.5 h.

<sup>b</sup> The results are obtained by GC with the internal standard method.

<sup>c</sup> The values of TOF (turnover frequency) are obtained from the formula:

TOF = the mole amount of reactant / (the mole amount of TEMPO · reaction time).

In the case of quaternary ammonium salts, the bromide salts are more useful than chloride salts in oxidation of benzyl alcohol, in which only a 61.7%, 64.6% or 38.9% yield of benzaldehyde is respectively obtained when TMAC, TEAC or TBAC is used as the additive (entries 1, 3, 5). While, 98.0%, 92.8% and 92.0% yields of benzaldehyde are obtained in the presence of TMAB, TEAB and TBAB (entries 2, 4,

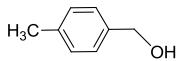
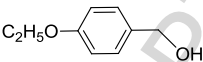
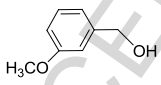
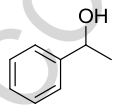
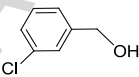
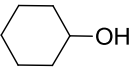
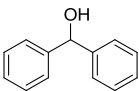
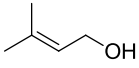
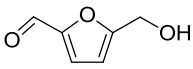
6). Otherwise, with DTAB as the additive, the yield of benzaldehyde is only 67.3% (entry 7), which showed that the long alkyl chain is disadvantageous to the alcohol oxidation in water. On the other hand, the influences of anionic surfactants are also examined. Therein, SDBS exhibits the outstanding promotion where the yield of benzaldehyde reaches 98.0%. However, the mediation of SDS is not too obvious, and only 48.4% yield of benzaldehyde is obtained. Moreover, the value of turnover frequency (TOF) arrives at 66 in the presence of several additives, which shows the suitable additive has obvious promotion effect on the oxidation of benzyl alcohol.

#### **3.4. The selective aerobic oxidation of different alcohols**

The oxidation of various alcohols is investigated with TEMPO-Ce(IV)-NaNO<sub>2</sub> catalytic system in aqueous media, and the results are listed in Table 3. In the oxidation of 4-methyl benzyl alcohol or 4-ethoxyl benzyl alcohol, 91.3% yield of 4-methyl benzaldehyde or 98.0% yield of 4-ethoxyl benzaldehyde is obtained, respectively (entries 1 and 2). Similarly, 3-methoxy benzyl alcohol is oxidized with a 98.0% yield of 3-methoxy benzaldehyde (entry 3), which shows that substituted position has no special effect for the electron donating groups. In case of phenyl ethanol, a 98.0 yield of acetophenone is obtained (entry 4). Furthermore, 3-chloro benzyl alcohol is also efficiently oxidized with a 95.9% yield of 3-chloro benzaldehyde (entry 5). These obtained results are comparable to the best literature data for the oxidation of different benzylic alcohols in aqueous media [30-32]. Besides, 55.6% yield of cyclohexanone or 45.5% yield of benzophenone is got from the oxidation of cyclohexanol or diphenylmethanol with this catalytic system (entries

6 and 7). A 58.2% yield of 3-methyl-2-butenal is obtained for the oxidation of 3-methyl-2-butenol (entry 8). These results showed that the naphthenic and aliphatic alcohols were more difficult than aromatic alcohols with TEMPO-CAN-NaNO<sub>2</sub> catalyst systems, in which the moderate yield of aldehydes were obtained. On the other hand, 5-hydroxymethylfurfural is oxidized with a 12.4% yield of furan-2,5-dicarbaldehyde (entry 9), which is much better than the oxidation with vanadyl phosphate as the catalyst in water [33, 34]. This shows a promising technique for the selective oxidation of heterocyclic alcohols.

**Table 3.** The aerobic oxidation of various alcohols in aqueous media <sup>a</sup>

Entry	Substrate	TEMPO (mol%)	Time (h)	Conv. (%) <sup>b</sup>	Sel. (%) <sup>c</sup>	Yield (%)
1		1	1.5	92.2	99.0	91.3
2		1	2	99.0	99.0	98.0
3		1	1.5	99.0	99.0	98.0
4		3	4	99.0	99.0	98.0
5		3	4	99.0	96.9	95.9
6		3	6	56.1	99.0	55.6
7		3	4	46.0	99.0	45.5
8		2	3	58.8	99.0	58.2
9		2	3	12.5	99.0	12.4

<sup>a</sup> Reaction conditions: 1.08 g benzyl alcohol, in 10 mL water, 5.0 mol% CAN and 10.0 mol% NaNO<sub>2</sub>, in the presence of 20.0 mol% TEAB, under 0.3 MPa of O<sub>2</sub>, temperature 80 °C.

<sup>b</sup> Conv. means the conversion of benzyl alcohol. The results are obtained by GC with internal standard method.

<sup>c</sup> Sel. Means the selectivity of benzaldehyde. The results are obtained by GC with internal standard technique.

#### 4. Conclusion

In summary, we have developed a new TEMPO-containing catalytic system which allows the aerobic oxidation of different alcohols to the corresponding aldehydes or ketones in aqueous media. The yields of corresponding aldehydes add up to 91.3-98.0% in the oxidation of benzyl alcohol and substituted benzyl alcohols. For the oxidation of naphthenic and aliphatic alcohols, 55.6-58.2% yields of aldehydes are obtained. This will generate a promising application strategy for green synthesis of the carbonyl compounds.

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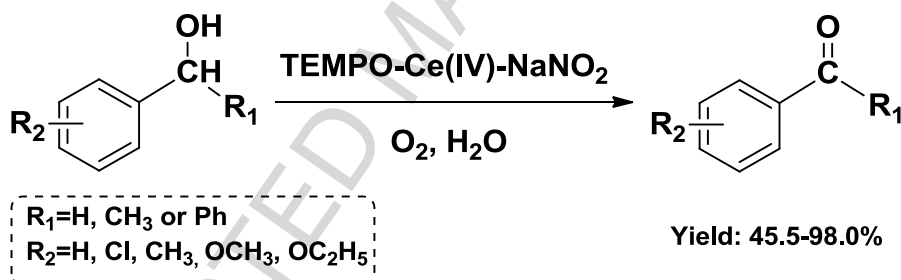
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## Graphical Abstract



An efficient and selective oxidation of different alcohols has been successfully performed for the synthesis of the corresponding aldehydes and ketones which is catalyzed by TEMPO-containing catalytic systems with molecular oxygen as the oxidant in aqueous media.



# Highly Efficient and Selective Aerobic Oxidation of Alcohols in Aqueous Media by TEMPO-Containing Catalytic Systems

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## Research Highlights

- A new catalytic system is developed for aerobic oxidation of alcohols.
- The efficient oxidation of different alcohols is achieved in aqueous media.
- The catalytic system is composed of TEMPO, Ce(IV) and NaNO<sub>2</sub>.
- 45.5-98.0% yields of aldehydes or ketones are obtained in the reaction.