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An effective oxidation of 2,3,6-trimethylphenol to 2,3,5-trimethylbenzoquinone using Fenton's reagent under mild conditions

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ABSTRACT

The oxidation of 2,3,6-trimethylphenol with Fenton's reagent has been investigated under mild conditions in aqueous phase. 2,3,5-Trimethylbenzoquinone was obtained with a selectivity of nearly 99.9% at 100% conversion of 2,3,6-trimethylphenol at 45 °C for 3 h. The different parameters like: the performance of different catalysts, the effect of catalyst concentration, reaction time, and reaction temperature has been investigated. Moreover, our new catalytic system was found also to be active in the oxidation of *o*-methylphenols to the corresponding benzoquinones by 5% H_2O_2 under mild conditions. In order to further study this reaction system, the possible mechanism was proposed. An important advantage of this catalytic system, apart from the organic solvent-free conditions, is that it is nontoxic, inexpensive and water-soluble.

1. Introduction

During recent years, in the wake of increasingly stringent environmental legislation, attention has been focused on the development of catalytic oxidations for the manufacture of fine chemicals [1]. The oxidation of phenols to quinones, for example, is a ubiquitous reaction in organic chemistry. These transformations are of great importance not only in the laboratory but also in industry, where they account for most of the applied processes in bulk and fine chemistry. Natural products having a benzoquinone structure also show biologically important properties such as cardiovascular, antitumour, antibacterial, antigerminative, and antiprotozoan activities [2–4]. Furthermore, benzoquinones are important fine chemicals in industry and are used to synthesize many pharmaceuticals [5]. For example, 2,3,5-trimethyl-1,4-benzoquinone (TMQ), as with a number of other benzoquinones, is an especially valuable starting material for the synthesis of V_E and other pharmaceuticals. The oxidation of 2,3,5-trimethylphenol (TMP) or 2,3,6-trimethylphenol is the key step for the production of 2,3,5-trimethylbenzoquinone. In industry, a current method of TMQ production is *para*-sulfonation of TMP, followed by oxidation with manganese dioxide. This method, however, is not suitable for continuable industrialized manufacture because of the contamination of inorganic salts and wastewater. Previously, various oxidation methods for trimethylphenol have been reported [6–14]. These different methods, however, show varying degrees of success as well as limitations due to the need for equimolar catalysts, high reaction temperatures, and the use of large quantities of organic solvents in reaction system with the possibility of explosion. Among these, $CuCl_2$ has been the most favored catalyst when used with an equimolar amount of TMP. Takehira [15] and Bodnar et al. [16] reduced the amount of the catalyst by employing nitrogen-containing organic compounds as co-catalysts. However, the co-catalysts were partly consumed in the reaction system. Recently, we reported that the catalyst $CuCo_2O_4$ showed high activity and selectivity in the oxidation of 2,3,5-trimethylphenol to 2,3,6-trimethylbenzoquinone with aqueous hydrogen peroxide at room temperature. However, the excessive use of H_2O_2 is unrealistic in an industrial process [17].

One hundred years ago, H.J.H. Fenton reported that ferrous ions strongly promote the oxidation of maleic acid by hydrogen peroxide [18]. Study has shown that the combination of H_2O_2 and a ferrous salt, "Fenton's reagent", is an efficient oxidant of a wide variety of organic substrates [19]. Subsequently Fenton's and like-Fenton's reagents were used in wastewater treatment under acidic conditions [20–24]. Although the use of Fenton's reagent

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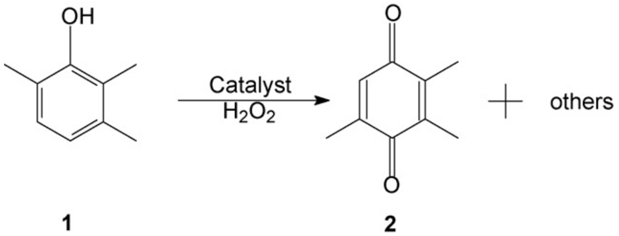


Scheme 1.

and related reactions are potentially useful oxidation processes for organic compounds in water, there are few reports in which Fenton's reagent was used for organic synthesis [25–28].

We report in this work a very simple, efficient and selective oxidation of 2,3,6-trimethylphenol (TMP) to 2,3,5-trimethylbenzoquinone with 5% H_2O_2 catalyzed by $FeSO_4$ (Fenton's reagent), developed under mild conditions. The reaction can take place in the aqueous phase and can be applied to the oxidation of *o*-methylphenols to the corresponding benzoquinones. As a water-soluble catalyst, $FeSO_4$ can be easily separated from the reaction products by simple disposal. In addition, the products are not contaminated with traces of metal catalyst.

2. Experimental

2.1. Materials

Methylphenols were donated by J&K Chemical Ltd. H_2O_2 (30%) and metal salts were analytical grade from Aldrich or Fluka.

2.2. General procedure for the oxidation of 2,3,6-trimethylphenol

The catalytic oxidation reactions of TMP were carried out using a 100 mL three-necked flask equipped with a reflux condenser, magnetic stirrer, super constant temperature oil bath and thermometer. In a typical reaction, TMP (1 mmol), $FeSO_4 \cdot 7H_2O$ (5 mol%) and deionized water (10 mL) were placed in the flask, and then 5% H_2O_2 (4.1 mmol) was added dropwise to the stirred mixture at 45 °C. Ten microlitres of fresh chloroform was added and then separated. The selectivities and conversions were determined by GC and GC–MS.

2.3. Physical measurements

GC analyses were performed using a gas chromatograph (GC-2010, Shimadzu) equipped with a flame ionization detector and a quartz capillary column (25 m × 0.3 mm) filled with Carbowax 20 M. GC–MS analyses of the products were carried out using a HP 5973/6890 system (electron impact ionization at 70 eV, He carrier gas, 30 m × 0.25 mm cross-linked 5% PHME siloxane (0.25 μm coating) capillary column; HP-5MS) or a VG-7070 instrument.

3. Results and discussion

3.1. Catalytic oxidation of 2,3,5-trimethylphenol

3.1.1. Performance of Fenton's and like-Fenton's reagents

The catalytic performance of Fenton's reagent and several prevailing like-Fenton's reagents for the oxidation of TMP by 5% H_2O_2 (Scheme 1) was compared under the same conditions with the results shown in Table 1. The oxidation of TMP (1 mmol) was carried out with 4.1 mol H_2O_2 (5%) as oxidant in the presence of a catalytic amount of various metal salts (5 mol%) in 10 mL deionized water at 45 °C within 3 h. As seen in Table 1, the reaction did not proceed in the absence of catalyst although autocatalysis by the reactor wall is often observed in oxidations [29]. We can also see that the selectivity of TMQ varied considerably although catalysts composed of the same metal ion and different anions (NO_3^-, SO_4^{2-} and Cl^-) yielded similar values of TMP conversion. On the other hand, different metal ions had a dramatic effect on the catalytic activity even when these catalysts have the same anions. It was found that the selectivity of TMQ decreased in the order $FeSO_4 \approx Fe_2(SO_4)_3 > Cr_2(SO_4)_3 > FeCl_2 \approx MnSO_4 > NiSO_4 > CuSO_4 > Fe(NO_3)_3 > Mn(NO_3)_2 > Co_2(SO_4)_3 > FeCl_3 > Cu(NO_3)_2 > Cr(NO_3)_3 > Ni(NO_3)_2 > Co(NO_3)_2$. At the same time the conversion of TMP reduced in the order $FeSO_4 \approx Fe_2(SO_4)_3 > Fe(NO_3)_3 \approx Co(NO_3)_2 > Co_2(SO_4)_3 > FeCl_2 \approx FeCl_3 > NiSO_4 \approx Ni(NO_3)_2 > CuSO_4 \approx Cu(NO_3)_2 > Mn(NO_3)_2 \approx MnSO_4 > Cr_2(SO_4)_3 \approx Cr(NO_3)_3$. It is interesting to point out that the $FeSO_4$ catalyst shows the highest conversion and selectivity in the same operating conditions. However, 4-chloro-2,3,5-trimethyphenol was detected when metal salts containing Cl^- were used as the catalyst. Among these, $FeCl_2$ and $FeCl_3$ yielded the most 4-chloro-2,3,5-trimethylphenol. The amount of 4-chloro-2,3,5-trimethyphenol clearly increased and the selectivity of TMQ was markedly reduced when $FeCl_2$ was replaced by $FeCl_3$. From Table 1, it was found that the catalytic activity of $FeCl_2$ was more effective than that of $FeCl_3$ [30], but both of them are lower than $FeSO_4$. Therefore, we suggest $FeSO_4$ (Fenton's reagent) as the most suitable catalyst.

Table 1
Oxidation of TMP with different catalysts.

Catalyst	Conversion (mol%)	Selectivity (mol%)	Isolated yield (mol%)
–	0.0	0.0	0.0
$Cr(NO_3)_3$	66.2	78.6	51.1
$Cr_2(SO_4)_3$	66.7	99.3	66.0
$Mn(NO_3)_2$	80.3	85.6	68.7
$MnSO_4$	80.2	98.2	78.7
$Fe(NO_3)_3$	98.7	88.6	87.4
$Fe_2(SO_4)_3$	100.0	99.9	99.7
$FeSO_4$	100.0	99.9	99.8
$FeCl_3$	96.3	84.3	81.1
$FeCl_2$	96.7	98.3	95.0
$Co(NO_3)_3$	98.5	60.5	59.5
$Co_2(SO_4)_3$	97.5	85.5	83.3
$Ni(NO_3)_2$	91.3	72.9	66.5
$NiSO_4$	91.7	92.5	84.6
$Cu(NO_3)_2$	83.6	80.5	62.2
$CuSO_4$	82.9	90.3	74.7

Reaction conditions: 1 mmol TMP, 5 mol% catalyst, 10 mL deionized water, 4.1 mol 5% H_2O_2, reaction time = 3 h, reaction temperature = 45 °C.

3.1.2. Effect of catalyst concentration

In order to lucubrating the catalytic performance of Fenton's reagent, the oxidative effect of TMQ by $FeSO_4$ was investigated using 4.1 mol 5% H_2O_2 as oxidant in 10 mL deionized water at 45 °C within 3 h. Fig. 1 displays the conversion of TMP and the selectivity of TMQ under the same conditions as a function of the $FeSO_4$ to TMP ratio. An initial steep increase in the conversion of TMP was observed when the amount of $FeSO_4$ was increased up to 5 mol%; beyond this amount, the conversion of TMP levelled off in the reaction system (up to 100%). The selectivity of TMQ was clearly reduced when the amount of $FeSO_4$ is greater than 5 mol%. Other products were detected by GC–MS when the amount of catalyst added was 8 mol%. Muconic acid, maleic acid and oxalic acid were found to be present in this reaction system. This point is in accordance with most of the literature [20,31–32]. Therefore, 5 mol% $FeSO_4$ would appear to be the optimum amount of catalyst for selectively producing TMQ.

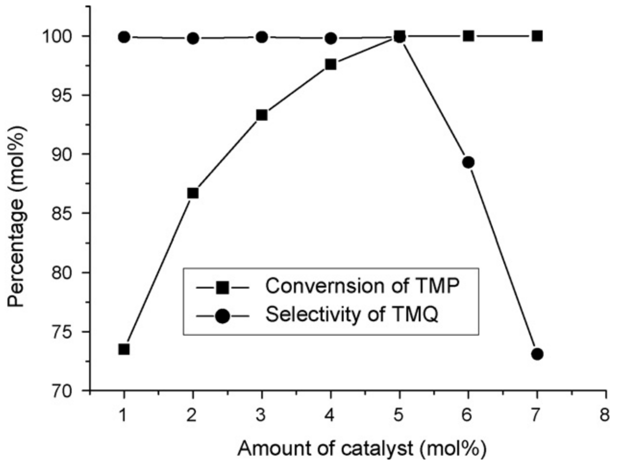


Fig. 1. The effect of the amount of catalyst (reaction conditions: 1 mmol TMP, catalyst = $FeSO_4$, 10 mL deionized water, 4.1 mol 5% H_2O_2, reaction time = 3 h, reaction temperature = 45 °C).

3.1.3. *Influence of reaction time*

Fig. 2 presents the species (including 2,3,6-trimethylphenol and 2,3,5-trimethylbenzoquinone) as a function of reaction time using an $FeSO_4$ catalyst, 4.1 mol 5% H_2O_2 as oxidant, and 10 mL deionized water as solvent at 45 °C. This shows that the rate of TMP conversion reaches a maximum (100%) after 3.0 h at which point the TMQ selectivity is 99.9%. With the increase of the reaction time, the TMQ selectivity decreased slightly because it was over oxidized by unreacted H_2O_2 to other products such as the hydroxylated p-benzoquinones at 3.5 h. In order to farther investigating the influences of reaction time, we extended the reaction time from 3.5 h to 5 h. Muconic acid, maleic acid, *etc.* were detected by GC–MS in this reaction system. We, therefore, suggest that 3 h is the most suitable reaction time at 45 °C. Moreover, kinetic data were collected by taking multiple GC–MS measurements during the course of 3 h reaction. An assumption of first-order kinetics was made and a plot of the $\mathrm{Ln}(C/C_0)$ vs. time is shown in Fig. 2. Good linear correlation ($R^2 = 0.93$) is observed suggesting that the reaction is first-order with respect to 2,3,6-trimethylphenol. These data were also fit to higher order kinetics (i.e., second and third), which resulted in poorer correlation (lower R^2 values).

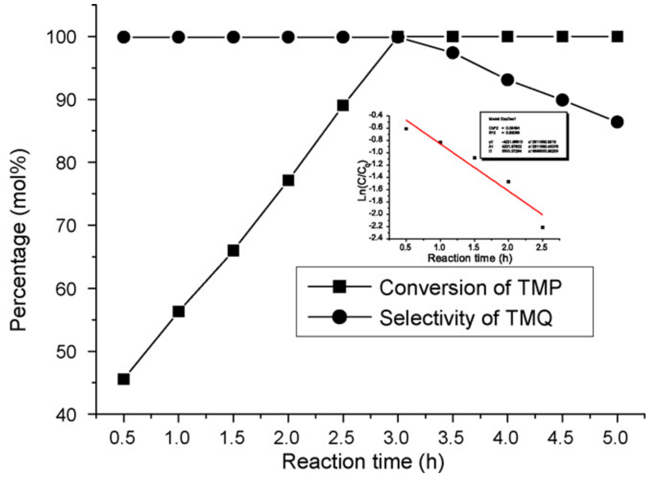


Fig. 2. The effect of reaction time (reaction conditions: 1 mmol TMP, mol 5% $FeSO_4$, 10 mL deionized water, 4.1 mol 5% H_2O_2, reaction temperature = 45 °C).

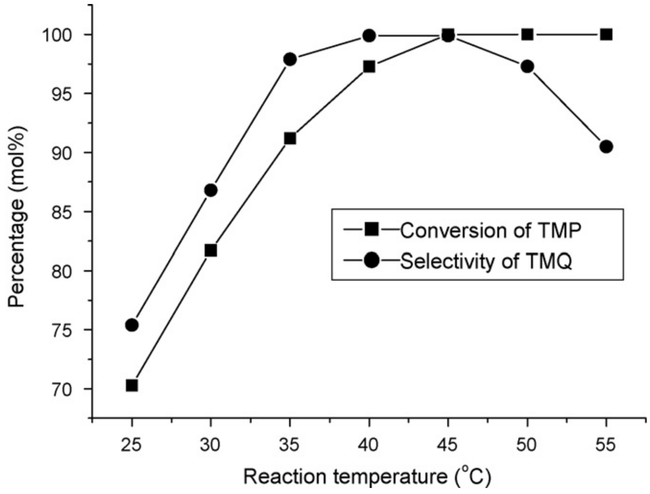


Fig. 3. The influence of reaction temperature (reaction conditions: 1 mmol TMP, mol 5% $FeSO_4$, 10 mL deionized water, 4.1 mol 5% H_2O_2).

3.1.4. *Effect of reaction temperature*

The dependence of the catalytic activity of $FeSO_4$ on the various reaction temperatures (25–55 °C) was investigated, and the results are shown in Fig. 3. The conversion of TMP and the selectivity of TMQ were found to increase with increasing reaction temperature when other conditions were not changed. The rate of TMP conversion levelled off at 45 °C and the selectivity of TMQ passed through a maximum at the same temperature. A further increase in the reaction temperature caused a decrease in the selectivity of TMQ because it was further oxidized, and some undesirable products, such as 2-hydroxyl-3,5,6-trimethylbenzoquinone and some ring-opening organic acids, were detected. On the other hand, when the temperature was lower than 45 °C a reduced selectivity for the desired product, 2,3,5-trimethyl-1,4-benzoquinone, was observed. The reason for the reduced selectivity can be explained by the formation of 2,2′,3,3′,5,5′-hexamethyl-4,4′-biphenol along with TMQ. These results indicate that the reaction temperature is a very important factor.

3.1.5. *Influence of H_2O_2*

Although aqueous H_2O_2 is generally regarded as an environmentally friendly “green” reagent [33–34] because it gives water as the sole by-product of oxidation, the strength and amount of hydrogen peroxide will affect the conversion of TMP and the selectivity of TMQ. We therefore investigated the influence of H_2O_2 (5–30 wt%) on this reaction system at 45 °C with the molar ratio of 2,3,5-trimethylbenzoquinone:H_2O_2 as 1:4.1 (Fig. 4). We found that 5% H_2O_2 gave nearly 100% conversion of TMP with >99% selective formation of TMQ. When we used other concentrations of H_2O_2, we observed that all selectivities of TMQ were lower than 5% H_2O_2, and selectivity of TMQ was reduced along with the increase of H_2O_2 concentration. Simultaneously, the total amount of side products increased (Fig. 4). In addition, the effect of the molar ratio of 2,3,5-trimethylphenol to H_2O_2 was also investigated (Fig. 5). Keeping the reaction temperature at 45 °C and using 5% H_2O_2 and 10 mL deionized water, we examined the effect of molar ratios of 2,3,5-trimethylphenol and 5% H_2O_2 between 1:2 and 1:4.5. In this reaction system, the selectivity of TMQ remained >99%, but the conversion of TMP increased from 66.7% to 100% when the molar ratio of 2,3,5-trimethylphenol to 5% H_2O_2 was increased from 1:2 to 1:4.1. A ratio greater than 1:4.1 was unnecessary.

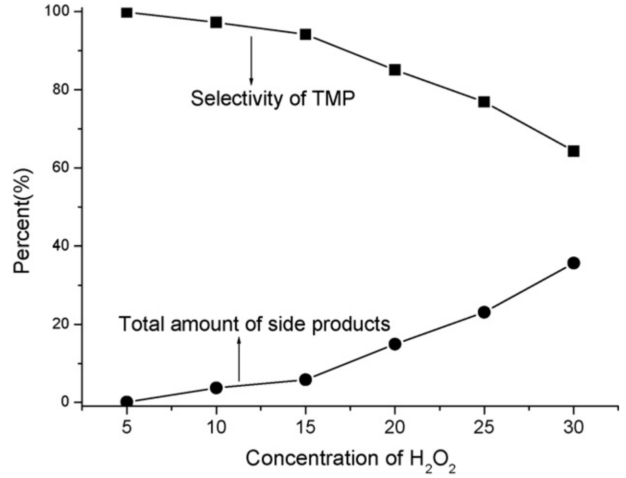


Fig. 4. The effect of H_2O_2 concentration (reaction conditions: 1 mmol TMP, 5 mol% catalyst, 10 mL deionized water, reaction time = 3 h, reaction temperature = 45 °C).

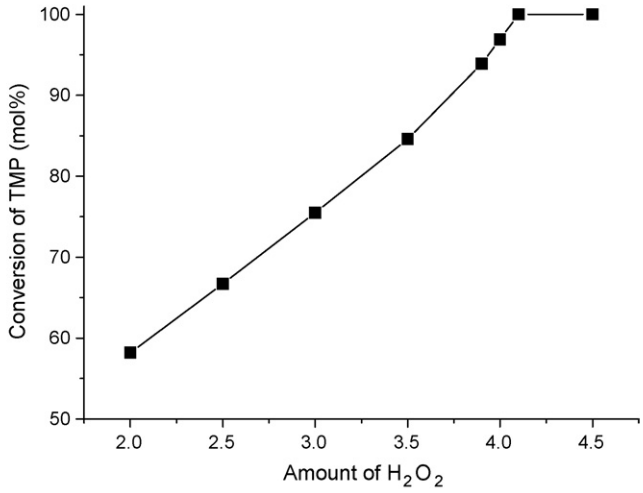
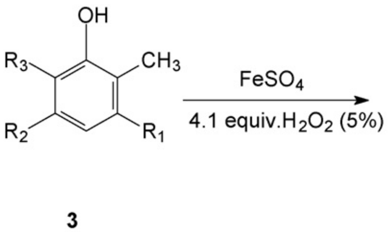


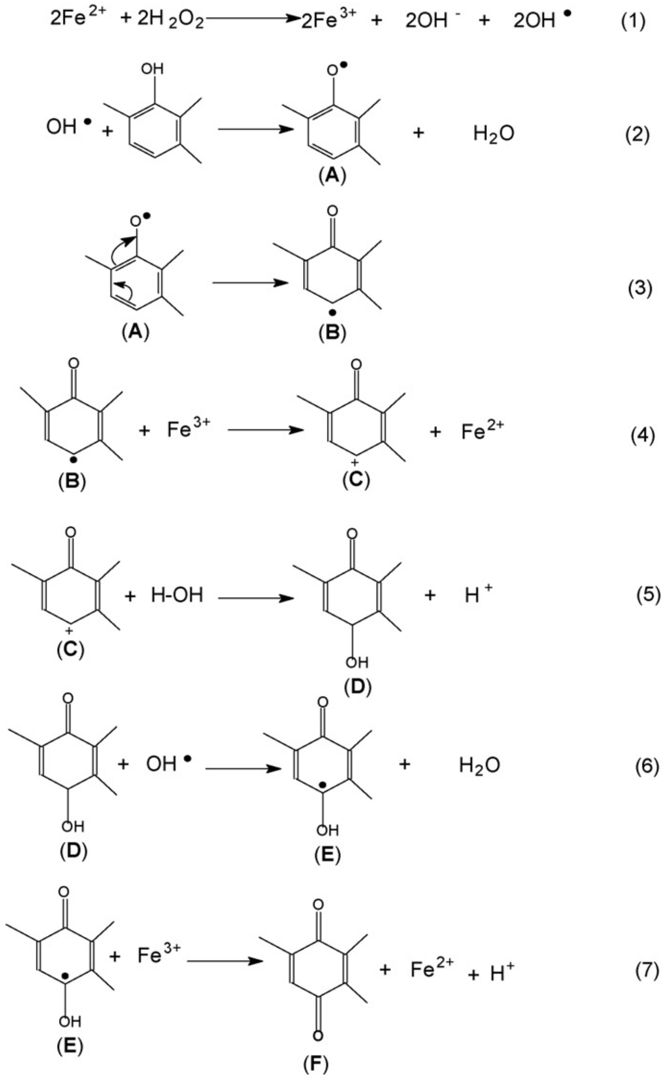
Fig. 5. The influence of the amount of H_2O_2 (reaction conditions: 1 mmol TMP, 5 mol% catalyst, 10 mL deionized water, reaction time = 3 h, reaction temperature = 45 °C).

3.2. The possible reaction mechanism

In order to extrapolating the present finding to other methylphenols oxidations. The possible was proposed in Scheme 3. The generally accepted free radical chain mechanism for the Fenton and like-Fenton reactions [19,35–37]. First, the Fe^{2+} was oxidized to the Fe^{3+} by hydrogen peroxide, and the hydroxyl radical was produced simultaneously (Eq. (1), Scheme 3). In the presence of 2,3,6-trimethylphenol, the hydroxyl radical entered into the hydroxyl of 2,3,6-trimethylphenol to abstract hydrogen, then the oxygenous radical (**A**, Eq. (2), Scheme 3) was presented. The oxygenous radical, however, favored to rearrange to the para position of **A** (**B**, Eq. (3), Scheme 3) [23,32]. The **B** is a tertiary radical, and adjacented to any group capable of stabilizing a positive charge [28,38], it allowed the starting oxidation state of iron to be re-established by an electro-transfer reaction (**C**, Eq. (4), Scheme 3), which was followed to be hydroxylated be water (**D**, Eq. (5), Scheme 3). The alcohol (**D**) was easy to react with hydroxyl radical [28] to produce the tertiary hydroxyalkyl radical stabilized by the adjoining alcoholic function (**E**, Eq. (6), Scheme 3). Finally, **E** would be react with Fe^{3+}, and 2,3,5-trimethylbenzoquinone was produced. From Scheme 3, we also found that Fe^{3+}/Fe^{2+} was a catalytic cycle neither more nor less than.



Scheme 2.



Scheme 3.

3.3. Oxidation of other methylphenols to methylbenzoquinones

Having established what appear to be the optimum conditions, and validating proposed reaction mechanism, we switched our attention to the substrate. A variety of substituted *o*-methylphenols were then reacted using this remarkably simple procedure (Scheme 2) with the results presented in Table 2. We found that all *o*-methylphenols were oxidized by Fenton's reagent with good selectivity and 100% conversion. At the same time, the selectivity of the products was affected by the number and position of substituted methyl groups. The reaction worked well with hin-

Table 2
Oxidation of *o*-methylphenols with $FeSO_4$–H_2O_2[a].

Entry	Substrate	R_1	R_2	R_3	Product	Selectivity (mol%)[b]
1[c]	3a	CH_3	CH_3	CH_3	4a	99.9
2	3b	CH_3	H	CH_3	4b	99.9
3	3c	CH_3	CH_3	H	4c	87.5
4	3d	H	H	CH_3	4d	73.3
5	3e	H	CH_3	H	4e	61.3
6	3f	CH_3	H	H	4f	81.6
7[d]	3g	H	H	H	4g	43.5

[a] Reaction conditions: 1 mmol substrate, 5% mol $FeSO_4$, 10 mL deionized water, 4.1 mmol 5% H_2O_2, reaction time = 3 h, reaction temperature = 45 °C.

[b] Products were identified by GC using authentic samples for comparison.

[c] Reaction time = 5.5 h, reaction temperature = 60 °C.

[d] Determined by GC–MS and GC.

dered phenols such as 2,3,5,6-tetramethylphenol (Entry 1, Table 2), but the 2,3,5,6-tetramethylphenol required longer reaction times and higher reaction temperature to give the same conversion rate as other substituted *o*-methylphenols. The selectivity of 2-methyl-1,4-benzoquinone (4 g, Entry 7, Table 2) was only 43.5% however; more 2-methylphenol was over oxidized to short-chain acids such as acetic oxalic and formic acids. In order to further investigating the reaction system, we select phenol as an oxidized substrate at the same time. Disappointedly, hardly benzoquinone occurred in the catalytic system. This result was similar with many Refs. [20,31,32,35–38].

4. Conclusion

In conclusion, Fenton's reagent, which is composed of $FeSO_4$ and aqueous hydrogen peroxide (5% H_2O_2), was found to be highly efficient for the selective oxidation of 2,3,6-trimethylphenol to the corresponding benzoquinone under mild conditions. Moreover, this new catalytic system was also active in the oxidation of *o*-methylphenols to the corresponding benzoquinones using 5% H_2O_2. An important advantage of this catalytic system, aside from the organic solvent-free conditions, is that it is nontoxic, inexpensive and water-soluble.

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