Fe (III) supported on resin as effective catalyst for the heterogeneous oxidation of phenol in aqueous solution

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Abstract

FeIII supported on resin as an effective catalyst for oxidation was prepared and applied for the degradation of aqueous phenol. Phenol was selected as a model pollutant and the catalytic oxidation was carried out in a batch reactor using hydrogen peroxide as the oxidant. The influent factors on oxidation, such as catalyst dosage, H2O2 concentration, pH, and phenol concentration were examined by considering both phenol conversion and chemical oxygen demand (COD) removal. The FeIII-resin catalyst possesses a high oxidation activity for phenol degradation in aqueous solution. The experimental results of this study show that almost 100% phenol conversion and over 80% COD removal can be achieved with the FeIII-resin catalyst catalytic oxidation system. A series of prepared resin were investigated for improving the oxidation efficiency. It was found that the reaction temperature and initial pH in solution significantly affected both of phenol conversion and COD removal efficiency. The activity of the catalyst significantly decreased at high pH, which was similar to the Fenton-like reaction mechanism. Results in this study indicate that the FeIII-resin catalytic oxidation process is an efficient method for the treatment of phenolic wastewater.

Keywords: FeIII-resin catalysts; Catalytic oxidation; Phenol; Hydrogen peroxide

1. Introduction

Phenol and its derivatives are used as raw materials in many petrochemical, chemical and pharmaceutical industries (Fajerweg and Debellefontaine, 1996). These toxic contaminants when present in water cause high toxicity in aqueous environment. For economic considerations, there are many wastewater treatment processes for phenolic compound removal, but there are few suitable processes to deal with this high toxicity effluent. The extraction method can be used if the concentration of phenol is above 1% in the effluent; however, economic considerations of solvent extraction limit this process (Miro et al., 1999; Greminger et al., 1982). On the other hand, biological methods often used to decompose phenolic effluents due to the bactericide properties of phenolic material (Clayton and Aydin, 1997; Fortuny et al., 1999). Wet air oxidation (WAO) is a general technology
for wastewater treatment of industrial effluent. Wet air oxidation is carried out at high temperature and high-pressure conditions with oxygen or air in aqueous systems to achieve the oxidation of organic compounds. To achieve a highly efficient oxidation in aqueous solution, the catalytic wet air oxidation (CWAO) method was developed and applied. In the last several decades, catalytic wet air oxidation has received the attention of many investigators for wastewater treatment. The CWAO process offers high efficiency for the degradation of phenolic compounds and yields harmless water and carbon dioxide as reaction products (Fortuny et al., 1998, 1999; Hamoudi et al., 1999; Hamoudi et al., 2000).

For the purpose of improving the oxidation efficiency and reducing the critical reaction conditions, catalytic wet peroxide oxidation process (CWPO) was applied for phenolic effluent treatment. Although the homogeneous catalyst applied in CWPO processes own a high efficiency, their recovery from the treated effluent is rather difficult and requires an additional process to remove the homogeneous catalyst in the reactor. One of the famous oxidation reactions is the Fenton’s process. The method for phenolic wastewater treatment with hydrogen peroxide is mostly based on homogeneous catalysis by Fe (II) ion in water, namely, Fenton-type reaction (Tang and Huang, 1996; Grigoropoulou and Philippopoulos, 1997; Grymonpre et al., 2001). The Fenton-type reaction used hydrogen peroxide as an oxidation agent and is also a supplier of hydroxyl radical. It is well known that the highly efficient oxidation of phenolic compounds can be obtained in this process. However, the use of FeII ion in aqueous could not be removed after the oxidation process and this metallic ion would become a new pollutant in the effluent. Therefore, the homogeneous catalytic oxidation should be modified to overcome the disadvantage of catalyst reuse in the catalytic oxidation process. Using a heterogeneous catalyst is a new approach to achieve a highly efficient oxidation process and provided an easy recovery process. Many catalytic wet oxidation (CWO) processes had been developed and applied for phenolic effluent treatment in many industries. The bimetallic catalyst was one of the most active fields in catalyst wet oxidation process. The use of inorganic metallic oxide catalysts for oxidation in aqueous solution has been applied to phenolic wastewater treatment (Barrault et al., 1998; Leyva et al., 1998; Yu et al., 1999; Hocevar et al., 2000; Phu et al., 2001; Wu et al., 2001; Dubey et al., 2002). Catalysts usually lose their activity because of leaching effects of metallic catalysts in acidic medium used in heterogeneous catalytic oxidation (Fajerweg and Debellefontaine, 1996; Barrault et al., 2000). The leaching effects of metallic catalysts and the deactivation of the catalyst are still challenges for developing advantageous catalyst for the oxidation of phenolic wastes. The previous reports showed that the activity of bimetallic catalyst was significantly influenced by the nature of metal (Chen et al., 2001; Ovejero et al., 2001; Kim and Ihm, 2002; Pirkanniemi and Sillanpää, 2002) composition of catalyst, fouling effect and dissolution metal of catalyst (Fajerweg and Debellefontaine, 1996; Barrault et al., 2000). However, based on the consideration of operating conditions and cost in many industrial effluent, those bimetallic catalyst was limited their application for many phenolic material and organic wastewater treatment.

The purpose of this investigation was to explore a new catalyst and to apply it for phenolic wastewater treatment by CWPO process. This study presents the effects of reaction properties on the activity of a FeIII-resin catalyst for catalytic wet peroxide oxidation process of phenolic aqueous solutions. The FeIII-resin catalyst was synthesized in this study and it was used to promote phenol decomposition with hydrogen peroxide in wastewater treatment. The influencing factors in catalytic wet peroxide oxidation are also discussed in detail.

2. Materials and methods

2.1. Materials and characterization

Phenol and FeCl3 (99%, purity) were purchased from MERCK Co. (Germany). The macroporous weak acidic cation exchange resin (C-160) was purchased from PUROLITE (ion exchange capacity: 3.0 meq/ml/min, United Kingdom). FeIII-resin catalysts were prepared from C-160 resin with various molar concentration of iron chloride in aqueous solution. Hydrogen peroxide (OSAKA Co., Japan, 35%) was used as the oxidant in solution. Fresh and used catalysts were characterized with respect to their adsorption isotherm and BET specific surface area using N2 physical adsorption at 77 K on an accelerated surface area and porosimetry system (ASAP 2010 Micromeritics Co.). Surface area and pore diameters of the catalysts were determined from the application of the BET and Dubinin–Radushkevich equation, respectively, to the N2 adsorption isotherm at relative pressures of between 0.02 and 0.06. Elemental analysis was carried out using a scanning electron microscope (JEOL-JXA-840) with EDAX detector (LINKSAN10000/85S).

2.2. Catalyst preparation and oxidation

The weak acidic cation exchange resin supports were pretreated with acetone 3–4 times, and then washed by deionized water to remove the interferences factors. They were soaked in different concentrations of ferric chloride in the aqueous solution for more than 24 h ($V_{\text{salt solution}} / V_{\text{resin}} = 10$). Then they were filtered by sieving and washed with deionized water. The resins sup-
porting FeCl₃ were baked at 373 K for 24 h. The CWPO process was carried out in a 0.6-l batch reactor. The wastewater was fed into the reactor at a fixed reaction temperature, which was fixed using a water bath. Fe-resin catalysts of a suitable dosage were added into the reactor. Then dosage of hydrogen peroxide was then introduced into the reactor. At the desired time interval thereafter, liquid sample was taken from the reactor and chemical oxygen demand (COD) was analyzed using the standard methods, Section 5220D, APHA, 1995. Sampling was continued for 180 min. The reaction was carried out in the 40–80°C temperature range, catalyst loading range of 0.5–5 g/l, phenol initial concentration range of 500–2000 mg/l, and hydrogen peroxide range of 0.05–0.3 mol/l. Blank experiments were carried out only using hydrogen peroxide and phenol in solution. The concentration of H₂O₂ in solution was measured by a self-indicating potassium permanganate titration. Portion of sample was transferred to a 250 ml flask, and 100 ml and 10 ml H₂SO₄ (2 M) were added. Solutions were titrated using standard potassium permanganate solution (0.02 M) until the first permanent pink color appeared (Cornish et al., 2000).

2.3. Analysis in solution

The phenol concentration during oxidation was determined by high-performance-liquid chromatography (HPLC) Jasco PU980 (Japan Spectroscopic, Tokyo, Japan) with a UV detector. A methanol: water (65:35, v:v) mobile phase was used at a flow rate of 1 ml/min in conjunction with 25 cm × 4.6 mm C-18 column. The HPLC was equipped with an ultraviolet detector at 540 nm.

3. Results and discussion

3.1. Effect of Fe loading on activity of catalyst

Fe³⁺-resin catalysts were prepared from ion exchange resin with different amounts of Fe loading. The pore properties and surface element analysis of Fe³⁺-resin is given in Table 1. It can be seen that the low pore volume and surface area of ion-exchange resin is found with different immersing iron concentration (0.1–0.3 M). The iron loading in resin were measured by using dissolution, SEM-EDS and ion exchange loading method. The ion exchange loading method was to proceed with measuring the iron concentration before and after mixing with the ion exchange resin. Those measurements were indicated that a higher concentration of iron content by preparing with higher measuring iron concentration. All the iron content tests evidenced this result. As showing in Table 1, the measurement the ion exchange loading method showed that the iron content was higher than the result of SEM-EDS analysis and dissolution test (data is shown in Table 1). Therefore, it was proposed that the loading iron in ion exchange resin was heterogeneous. Otherwise, the low concentration of dissolution iron ion indicated that exchanged iron in resin did not completely dissolve even in 0.5 M HCl solution. The surface area of Fe³⁺-resins slightly increased with increasing immersing salt concentration. The slight increase in surface area of exchanged resin may be due to the precipitates of exchange salt in resin. However, it can be found that the surface area is still very low even immersing at high concentration salt solution. In order to evaluate the effect iron loading and surface area on the oxidation efficiency, the oxidations of phenol by Fe³⁺-resin catalyst were carried out in a stirred reactor with 0.1 M (mol/l) H₂O₂ and 3 g Fe³⁺-resin catalyst at 80°C. The profiles of residual phenol concentration and COD with various catalysts are shown in Fig. 1(a) and (b). As can be seen in Fig. 1(a) and (b), both residual phenol concentration and COD remained constant in absence of the resin-catalyst with 0.1 M H₂O₂ after 2h of oxidation. It was indicated that phenol did not degrade even in the presence of 0.1 M hydrogen peroxide in the solution. In the present of the Fe³⁺-resin catalysts, the residual phenol concentration rapidly decreased to less than 30 mg/l after 40 min and over 75% COD removal could be achieved. Different amount of Fe loading in catalyst (M1 Fe³⁺-resin and M3 Fe³⁺-resin) showed the same oxidation activity. The same activity of Fe³⁺-resin with various amount of Fe loading in resin was due to the

<table>
<thead>
<tr>
<th>Sample</th>
<th>Fe contentᵃ (mg/g resin)</th>
<th>Fe contentᵇ (mg/g resin)</th>
<th>Fe contentᶜ (mg/g resin)</th>
<th>Surface area, S_{BET} (m²/g)</th>
<th>Median pore diameter Å</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resin</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.24</td>
<td>18.29</td>
</tr>
<tr>
<td>M1 Fe-resin</td>
<td>477</td>
<td>275</td>
<td>25</td>
<td>0.70</td>
<td>11.95</td>
</tr>
<tr>
<td>M2 Fe-resin</td>
<td>558</td>
<td>529</td>
<td>33</td>
<td>3.01</td>
<td>11.86</td>
</tr>
<tr>
<td>M3 Fe-resin</td>
<td>616</td>
<td>556</td>
<td>36</td>
<td>9.22</td>
<td>11.62</td>
</tr>
</tbody>
</table>

ᵃ Measured by SEM-EDS on the surface of Fe-resin catalyst.
ᵇ Measured the iron concentration before and after mixing with the of resin.
ᶜ Dissolution test in 0.5 M HCl.
same Fe content in the resin by various preparation processes. The iron loading was also evidenced in Table 1.

On the other hand, it can be seen that the conversion efficiency of phenol was always higher than the efficiency of COD removal during the period of oxidation. It is possible that the intermediates in oxidation did not completely convert to the final products (H₂O and CO₂). Partial oxidation products of phenol were found in many previous reports on the wet oxidation process (Eftaxias et al., 2001). Therefore, the residual COD in the oxidation may be due to the partial oxidation products, which were incompletely oxidized by oxidant during the experiments. Fig. 2 indicates that COD rapidly decreased over the first 40 min and then leveled off. However, the phenol oxidation profile showed that complete phenol conversion was achieved after 20 min of

3.2. Influence of H₂O₂ concentration

The effect of H₂O₂ concentrations on phenol conversion and COD removal was tested for understanding the oxidation efficiency of phenol. The effect of H₂O₂ concentration on phenol conversion is shown in Fig. 3(a). It can be seen that the residual phenol concentration de-
increased with increasing H$_2$O$_2$ concentration in solution. It is interesting to note that the increase in H$_2$O$_2$ concentration up to 0.05 M (mol/l) results in over 90% phenol conversion. While H$_2$O$_2$ concentrations of up to 0.1 M (mol/l) result in over 99% phenol conversion achieved in 30 min. This result indicates that the concentration of H$_2$O$_2$ is strongly proportional to the phenol conversion. This result is the similar to a previous report (Chou and Huang, 1999) in CWPO system for phenolic wastewater treatment. They found that the decomposition rate of H$_2$O$_2$ was proportional to both H$_2$O$_2$ and catalyst concentration in the reactor. On the other hand, phenol conversion is also controlled by the competition between the rate of phenol and H$_2$O$_2$ decomposition.

Ilisz et al. (1999) showed that the decomposition rate of phenol is slower than the rate of HO$^\cdot$ radical formation. They also suggested that large amounts of HO$^\cdot$ radical decomposed from H$_2$O$_2$ cannot benefit the complete oxidation of phenol because of the shorter lifetime of HO$^\cdot$ free radical in the solution. If a large amount of HO$^\cdot$ radical decomposed from H$_2$O$_2$ in the first stage, the amount of H$_2$O$_2$ would be rapidly consumed in the first oxidation stage and hence phenol would not completely oxidize at stoichiometric dosages of H$_2$O$_2$.

In Fig. 3(b), it can be seen that the residual COD decreased with increasing H$_2$O$_2$ concentration but the final COD of the solutions almost had the same value at high H$_2$O$_2$ concentration (0.05 and 0.1 M). The residual COD from 0.01 M H$_2$O$_2$ was much higher than the residual COD from 0.05 and 0.1 M H$_2$O$_2$. The concentration with 0.01 M H$_2$O$_2$ is much lower than stoichiometric dosage of oxidant. It was indicated that the H$_2$O$_2$ dosage cannot completely oxide the phenol and intermediates to form the final product. Therefore, it can be seen that the high phenol conversion at low oxidant dosage and the low residual COD was obtained in the oxidation. Therefore, a poor COD removal can be expected at low H$_2$O$_2$ concentrations. It was suggested that complete oxidation of phenol and intermediate products should be related to the residual H$_2$O$_2$ concentration. It is interesting to note that the increase in H$_2$O$_2$ concentration, up to 0.1 M, cannot further increase the COD removal. It was due to the excess H$_2$O$_2$ reacts with the hydroxyl radical to form water and another product (Miller and Valentine, 1995). Therefore, it is proposed that the intermediate products and inefficient scavenging reactions at higher H$_2$O$_2$ concentration result and the final COD value of phenol solution cannot be effectively decreased. The yield of intermediate products would be reduced at the end of oxidation with the suitable dosage of H$_2$O$_2$ in the solution. Similar observations were also made by Dubey et al. (2002).

3.3. Influence of catalyst dosage

The effect of catalyst loading on the activity of Fe$^{III}$-resin catalyst during oxidation was investigated by varying the amount of catalyst in the range of 0.5–5 g in 600 ml of solution. The effect of catalyst loading on phenol removal during oxidation is presented in Fig. 4(a). The phenol conversion rate increased with increasing Fe$^{III}$-resin catalyst dosage in the solution. Other metal oxide catalysts (Yu et al., 1999) have shown the similar activity in advanced oxidation process for phenol wastewater treatment. Results indicated that additional amounts of catalyst to the reaction presented an enhanced effect on phenol conversion. The enhancement due to catalyst addition is proposed to the increase in efficiency of the decomposition rate of hydrogen peroxide. Therefore, high rates of oxidation of organic
materials with increasing the catalyst dosage can be expected based on the results of this work. The decomposition rate of organic compounds is proportional to the decrease of COD. Therefore, an evaluation of COD during oxidation is helpful to clarify the above proposal.

The effect of catalyst dosage on the COD removal during oxidation is shown in Fig. 4 (b). The decrease of COD strongly depends on the catalyst dosage. This decrease in COD also correlates with the enhancement of catalyst on the oxidation rate of organic material.

As shown in Fig. 4(a), phenol completely decomposed after 50 min of oxidation with a dosage of 0.5 g catalyst in the reactor. While the catalyst loading was higher than 0.5 g, over 95% phenol conversion was achieved with less time in the reactor. Therefore, oxidation rate increased with the catalyst loading in the reactor. Catalyst loading greater than 0.5 g in the reactor yields equivalent phenol conversion rates during the oxidation. This is due to the competition between OH• free radical formation and phenol oxidation in the reactor. While the rate of OH• free radical formation was greater than the phenol oxidation rate, phenol decomposition was the rate limiting step in oxidation. The oxidation rate was dominated by phenol oxidation rather free radical formation. Therefore, the increase in decomposition rate of hydrogen peroxide cannot enhance COD or phenol removal.

3.4. Effect of initial pH on activity of catalyst

The Fenton’s type reaction rate shows a strong dependence on pH during oxidation (Tang and Huang, 1996). They reported that the high acidity in oxidation, pH = 3–5, is enhances free radical formation and organic material oxidation. Valange et al. (1999) reported that the acidity of solution strongly influenced the formation rate of OH• radical. In this study, the Fenton’s like reaction was proposed in FeIII-resin catalyst/H2O2 system. The hydroxyl radical production significantly depends on pH. The conversion of phenol contributed to the decomposition of hydrogen peroxide, which contributes to the formation rate of OH• free radical. Fig. 5(a) shows the relationship between the residual phenol concentration and the initial pH during oxidation. It can be seen that the activity of FeIII-resin catalyst showed a strong dependence on the initial pH value. On the other hand, Fig. 5 (b) shows the relationship between the residual COD and initial pH value during oxidation. The COD value rapidly corresponding with the rate of phenol conversion. It is interesting to note that the removal both of phenol and COD is poor at high initial pH values. Fenton’s type reactions show the same decrease in phenol concentration and COD removal at higher pH condition in many previous investigations (Tang and Huang, 1996; Grigoropoulou and Philippopoulos, 1997; Barrault et al., 1998). Low initial pH (in acidic solution) supports free radical formation and promotes the oxidation of organic materials. Based on the observation of the effect of initial pH on the residual phenol concentration and COD removal, it was implied that a similar reaction might be carried out in the FeIII-resin catalyst system. It was indicated that the initial pH of FeIII-resin catalyst system showed a significant influence on the removal of phenol and COD. The possible reason is that the decrease in acidity of FeIII-resin catalytic oxidation benefited the formation rate of OH• free radical and enhanced the decomposition of organic compounds. It can be concluded that the initial pH in solution dominated the phenol conversion and oxidation rate of organic materials.

![Fig. 4](image-url)
The influence of pollutant concentration in the feed on the residual concentration of phenol is shown in Fig. 6(a). It can be seen that over 95% phenol conversion was achieved with increasing the phenol concentration up to 2000mg/l in the reactor. It is interesting to note that the activity of resin catalyst did not lose its activity upon increasing the pollutant concentration. It has been reported (Chou et al., 2001; Dubey et al., 2002) that high concentrations of organic pollutants usually inhibit free radicals to oxidize the partial oxidation products in the CWPO process. Therefore, the pollutant/catalyst ratio should be set at a suitable value to prevent the deactivation of free radicals in oxidation processes. Otherwise, the inhabitation of free radical formation at the active catalyst site stops the free radical reaction in oxidation. The influence of phenol concentration in feed on the COD removal in solution is shown in Fig. 6(b). It can also be seen that the decrease in COD removal was similar to the decrease in residual phenol concentration with different pollutant concentration. Based on the results in Figs. 6(a) and 6(b), it is implied that the COD removal was influenced in the concentration range of 500–2000mg/l. It is shown that the relative activity did not decrease with increasing the pollutant concentration during oxidation and that the inhibition of free radical observed on the surface of catalyst is not important while the pollutant concentration is up to 2000mg/l.

3.5. Influence of pollutant concentration

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3.6. Influence of reaction temperature

As shown in Fig. 7(a), phenol oxidation was carried out with Fe$^{III}$-resin in the temperature range from 40 to 80 °C. It can be seen that the residual phenol concentration rapidly decreased at 60 and 80 °C but less changed at 40 °C. The effect of reaction temperature on the COD removal is shown in Fig. 7(b). The COD removal showed a minimal decrease at low reaction temperature. It is well known that oxidation rate and COD removal are strongly dependent on the OH$^-$ contribution in the Fenton-like reaction. The free radical concentration usually increases with increasing reaction temperature in CWPO systems. However, the higher reaction temperature could enhance the thermal degradation rate of H$_2$O$_2$. Therefore, the removal of COD (or the oxidation rate of organic material) should be function of the competition between HO$^-$ radical formation and thermal degradation of hydrogen peroxide. A previous report (Dubey et al., 2002) also revealed a similar conclusion in CWPO systems. It was reported that the phenol conversion could be related to the competition between thermal decomposition and free radical formation. Based on the results of this study, it is indicated that the formation rate of free radical was greater than the thermal decomposition of H$_2$O$_2$ at high temperatures (60–80 °C). This viewpoint can be also be evidenced by the phenol conversion rate and decrease in COD removal at higher reaction temperatures.

4. Conclusion

The ion-exchange resin as the support of Fe$^{III}$ catalysts shows a high activity to oxidize high concentrations of phenol in aqueous solution. Over 95% phenol removal and 75% COD removal can be achieved by using a Fe$^{III}$-resin catalyst process. It is shown that the phenol conversion and COD removal strongly depend on the dosage of catalyst, hydrogen peroxide and initial pH. The Fenton’s like reaction might exist in the Fe$^{III}$-resin catalyst system. The oxidation rate of Fe$^{III}$-resin catalyst system is dominated by the formation of OH$^-$ radical and shows a significant influence on phenol decomposition. The activity of catalyst does not change with increasing the pollutant concentration in the range of 500–2000 mg/l. Acidic environments of Fe-resin catalytic oxidation benefit from the formation rate of OH$^-$ radical and enhanced the decomposition of organic compounds. Therefore, the initial pH in solution dominated the phenol conversion and oxidation rate of organic material. The reaction temperature affects formation rate of free radical and the rate of thermal decomposition of H$_2$O$_2$. A suitable reaction temperature range must be used in Fe$^{III}$-resin catalyst systems. High efficiency oxidation of phenol was achieved in this study and it was also worth applying this catalytic for another highly toxicity materials treatment in wastewater.

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References


