

Degradation of 2-Naphthalenesulfonate in Aqueous Solution by Hydrogen Peroxide in the Presence of Basic Oxygen Furnace Slag

Chyow-San Chiou^[1], Chiung-Fen Chang^[2], Ching-Yuan Chang^[3]

Graduate Institute of Environmental Engineering, National Taiwan University
Taipei, Taiwan 106, R.O.C.

Chang-Tang Chang^[4], and Yuan-Shan Li^[5]

Department of Environmental Engineering, National I-Lan University
I-Lan, Taiwan 260, R.O.C.

Abstract—Basic oxygen furnace slag (BOF slag) is a final waste in the steel making process, which contains 12.5 and 4.5 wt% of FeO and Fe₂O₃, respectively. The abundant iron-containing property of BOF slag makes it a possible catalyst for Fenton's reactions. This study evaluated the performance of H₂O₂ with BOF slag denoted as H₂O₂/BOF slag process to degrade 2-naphthalenesulfonate (2-NS). The concentration of total organic carbon (TOC) was chosen as a mineralization index of the degradation of 2-NS by the H₂O₂/BOF slag process. The experimental results revealed that the adequate pH values for better mineralization of 2-NS were found to be below 3. An optimum mineralization efficiency of 2-NS was evaluated as the BOF slag loading and the initial concentration of H₂O₂ were 15 g/L and 700 mg/L, respectively.

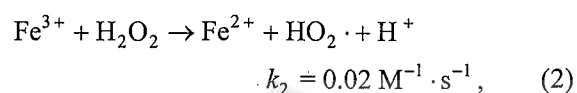
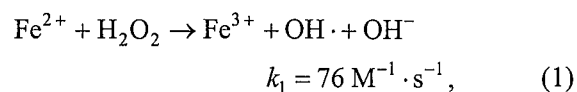
Key Words : Basic oxygen furnace slag, Fenton's reaction, 2-Naphthalenesulfonate, Mineralization

INTRODUCTION

Naphthalenesulfonic acids (NSAs) are important dye intermediates in the textile industry using many azo dyes and pigments (Storm *et al.*, 1999). The 2-naphthalenesulfonic acid (2-NSA), one of the NSAs, has been commonly used as a flotation collector, initiator for the catalytic polymerization of caprolactam, and tanning materials (Alonso and Barcelo, 1999). Its salt (2-naphthalenesulfonate, 2-NS) has been also used as a brightening and stabilization agent in the electroplating solution of printed wiring board (PWB) industry (Fang, 1996). Due to the resistance to microbial degradation, aromatic sulfonates are difficult to be treated by biological process (Sorensen and Frimmel, 1997). Furthermore, due to the low octanol-water partition coefficients (log *K_{OW}* values) (Schwarzenbach *et al.*, 1993), aromatic sulfonates can move with the aquatic system. Consequently, aromatic sulfonates can be found from the effluent of traditional wastewater treatment plant as well as surface water. Therefore, the treatment of 2-NS is of considerable importance in envi-

ronmental protection.

The 2-NS, being to be persistent to microbial degradation, had been successfully treated by the chemical oxidation ozonation combined with UV radiation (Chen *et al.*, 2002). Recently, the application of the Fenton's reactions to produce hydroxyl radicals to degrade organic pollutants has attracted extensive attention (Walling, 1975). It has been successfully applied to degrade aromatic compounds (Saxe *et al.*, 2000; Rivas *et al.*, 2001; Wang and Lemley, 2001). Through the agent of Fenton's reactions, which is a mixture of H₂O₂ with iron salts, a strong oxidizing hydroxyl radical, OH·, can be generated according to the following reactions (Chen and Pignatello, 1997; Tuba and Gurol, 2002).



[1] 邱求三, To whom all correspondence should be addressed

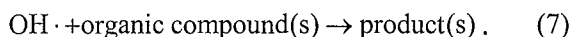
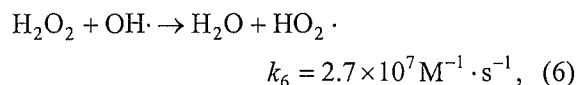
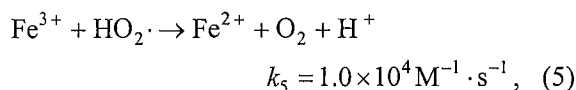
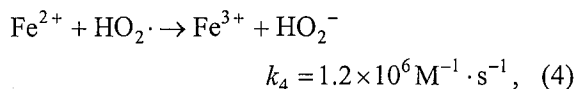
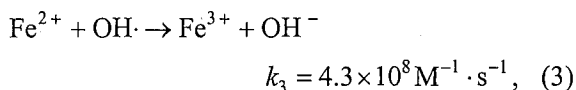
[2] 張瓊芬

[3] 張慶源

[4] 張章堂

[5] 李元陞





The application of Fenton's reactions has been limited by the slurry generation in which the precipitate of ferric hydroxide requires additional separation and disposal. In order to reduce the inconvenience, the use of iron oxide as a catalyst to enhance the oxidization of organic contaminants with hydrogen peroxide has been recently employed (Watts *et al.*, 1994; Lin and Gurol, 1998). Watts *et al.* (1997) applied $\alpha\text{-Fe}_2\text{O}_3$ in the oxidation of chlorobenzenes. Chou *et al.* (2001) reported the oxidation of benzoic acid using H_2O_2 with a novel supported $\gamma\text{-FeOOH}$ catalyst in a circulating fluidized-bed reactor.

In the steel making process, the molten iron is converted into steel with oxygen in basic oxygen furnace (BOF). The slag formed in the BOF after being solidified is called BOF slag, which is a gray, angular and porous chunk (China Steel Corp., 1994). The major constituents of BOF slag are CaO , SiO_2 , Fe_2O_3 , FeO , MgO , and MnO . It contains about 12.5 and 4.5 wt% of FeO and Fe_2O_3 , respectively. The abundant iron-containing property of BOF slag makes it a potential catalyst for Fenton's reactions (Li, 1999).

This study assessed the performance of BOF slag in enhancing H_2O_2 to degrade 2-NS. The concentrations of 2-NS were analyzed at specified time intervals to determine the degradation rate of 2-NS. Moreover, the concentration of total organic carbon (TOC) was chosen as a mineralization index of degradation of 2-NS by the process. The effects of pH value, H_2O_2 concentration, BOF slag concentration and initial concentration of 2-NS on the oxidation of 2-NS were examined.

MATERIALS AND METHODS

2-NS ($\text{C}_{10}\text{H}_7\text{SO}_3\text{NaO}$, FW = 230.22) with high quality was purchased from Aldrich (Milwaukee, WI,

U.S.A.) and used without any further purification. Hydrogen peroxide (35%) was supplied by Shimakyu Co. (Japan). BOF slag was obtained from China Steel Corp., Taiwan. The pretreatment of the BOF slag comprised several steps. Firstly, the BOF slag was washed by distilled water to remove the crushed and adhering dust. Secondly, it was dried at 383K in an oven and then stored in the desiccator. Finally, BOF slag was sieved between 10 and 18 mesh sieves and that of mesh 10-18 was used as the iron source in this study. The TOC of sample was analyzed by a TOC analyzer (Tekmar Dohrmann Phoenix 8000). The mineralization efficiency of 2-NS ($\eta_{\text{TOC,NS}}$) is $(\text{TOC}_{\text{NS0}} - \text{TOC}_{\text{NS}})/\text{TOC}_{\text{NS0}}$. The titanium (IV) chloride method was used to determine the concentration of unreacted hydrogen peroxide in the reaction solution (Boltz and Howell, 1978). Concentration of dissolved ferrous iron was determined spectrophotometrically by the phenanthroline method. Total iron concentration was determined by an atomic absorbance spectrophotometer.

The batch experiments of oxidation reaction were conducted in a 3 L well-mixed round-bottle glass flask with water jacket. Mixing was provided by a variable speed motor connected to a stainless steel shaft. A pH controller was used to control pH value of solution during the whole reaction time by adding 0.1 M HNO_3 or NaOH solution into the reactor. Five various initial concentrations (C_{NS0}) of 2-NS wastewater (30, 50, 100, 150, and 200 mg/L) with 350 mg/L hydrogen peroxide and 10 g/L BOF slag at pH value of 2.0 ± 0.2 were tested to assess the effect of C_{NS0} . The effect of pH value of solution during the whole reaction time was studied at various pH values of 2.0, 3.0, 4.0, and 5.0 ± 0.2 with 350 mg/L hydrogen peroxide, 10 g/L BOF slag and 100 mg/L 2-NS. The influence of hydrogen peroxide was examined by dosing various hydrogen peroxide concentrations (180, 350, 700, 1050, 1440, and 1750 mg/L) into the reactor with 10 g/L BOF slag and 100 mg/L 2-NS at pH value of 2. The effect of BOF slag was investigated at various concentrations of 3, 5, 10, 15, and 20 g/L with 350 mg/L hydrogen peroxide and 100 mg/L 2-NS at pH value of 2. The temperatures of the above experiments were maintained at 25°C .

RESULTS AND DISCUSSION

Dissolution of iron from BOF slag

BOF slag contains about 12.5 and 4.5 wt% of FeO and Fe_2O_3 , respectively. In an acid solution, total iron ions are generated from the dissociation of FeO and Fe_2O_3 . The leaching rate of ferrous ion and total iron ion can be expressed by following equations:

$$\frac{dC_{Fe, aq}}{dt} = k_{Fe}^0 C_{BOF}, \quad (8)$$

$$\frac{dC_{Fe^{2+}}}{dt} = a \left(\frac{dC_{Fe, aq}}{dt} \right) = a k_{Fe}^0 C_{BOF}. \quad (9)$$

The amount of BOF slag in the solution is denoted as C_{BOF} . The concentrations of ferrous ion (Fe^{2+}) and total iron ion (Fe_{aq}) in aqueous solution are expressed as $C_{Fe^{2+}}$ and $C_{Fe, aq}$, respectively. k_{Fe}^0 is the leaching rate constant of total iron from BOF slag, and a is a constant relating the formation rate of Fe^{2+} and Fe_{aq} .

The variations of the leached concentrations of Fe^{2+} from the BOF slag with time at various pH values are shown in Fig. 1. As shown in this figure, the slopes of the linear regression result at pH = 2, 2.5, and 3 are about 1.52, 0.48, and 0.15, respectively. The results show the formation rate of Fe^{2+} at pH = 2 is an order of magnitude higher than that at pH = 3, indicating that the lower pH can enhance the leaching rate of Fe^{2+} from the BOF slag. CaO is one of the major components of BOF slag, and caused the basic character of BOF slag. It will consume a lot of acids to adjust the pH value of BOF slag solution lower than 2. In addition, there is an acid error of pH meter at lower pH value. So, the pH value of solution was kept at 2 for the following experiments.

The time variations of $C_{Fe^{2+}}$ and $C_{Fe, aq}$ at various amount of slag for pH = 2 are illustrated in Figs. 2(a) and 2(b), respectively. Good linear relationships were observed. Both $C_{Fe^{2+}}$ and $C_{Fe, aq}$ increased with the increasing amount of BOF slag. The plots of the slopes, which were obtained from Figs. 2(a) and 2(b), versus the amount of BOF slag are presented in Figs. 3(a) and 3(b), respectively. The rate constants, ak_{Fe}^0 and k_{Fe}^0 , can be obtained from the slopes of the linear fits from each figure as 0.1005 and 0.1628, respectively. The difference of values between ak_{Fe}^0 and k_{Fe}^0 can be attributed to the contribution of iron ions in solution other than the Fe^{2+} form.

Figures 4(a) and 4(b) present the effects of H_2O_2 and 2-NS on the leached concentrations of $C_{Fe, aq}$ and $C_{Fe^{2+}}$ from BOF slag, respectively. The experiments were conducted at pH = 2.0 ± 0.2 in the presence of three different conditions: (A) with 10 g/L BOF slag only, (B) 10 g/L BOF slag with 350 mg/L H_2O_2 , and (C) 10 g/L BOF slag with 350 mg/L H_2O_2 and 100 mg/L 2-NS. As shown in Fig. 4(a), the concentration of $C_{Fe, aq}$ is linear proportional to reaction time with no significant difference in these three conditions.

However, there is an obvious difference in $C_{Fe^{2+}}$ for these three conditions as shown in Fig. 4(b). In condition A, which contains only BOF slag in the solution, the ferrous ion concentration increased

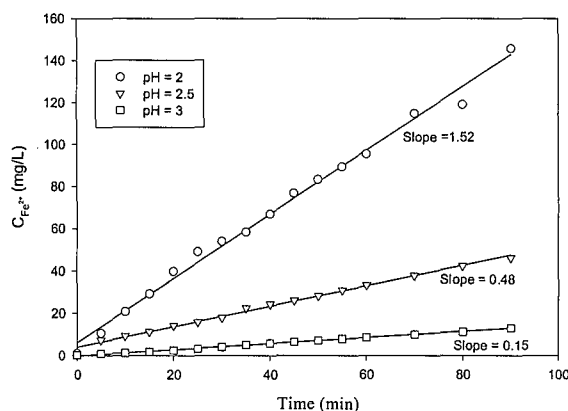


Fig. 1. Time variation of leached Fe^{2+} concentration ($C_{Fe^{2+}}$) at various pH values. Experimental conditions: 20 g/L BOF slag, temperature (T) at 25°C. \circ , ∇ , \square : pH = 2, 2.5, 3. The pH of solution was adjusted by HNO_3 . The R^2 values of regression lines of \circ , ∇ , \square : 0.993, 0.993, 0.997.

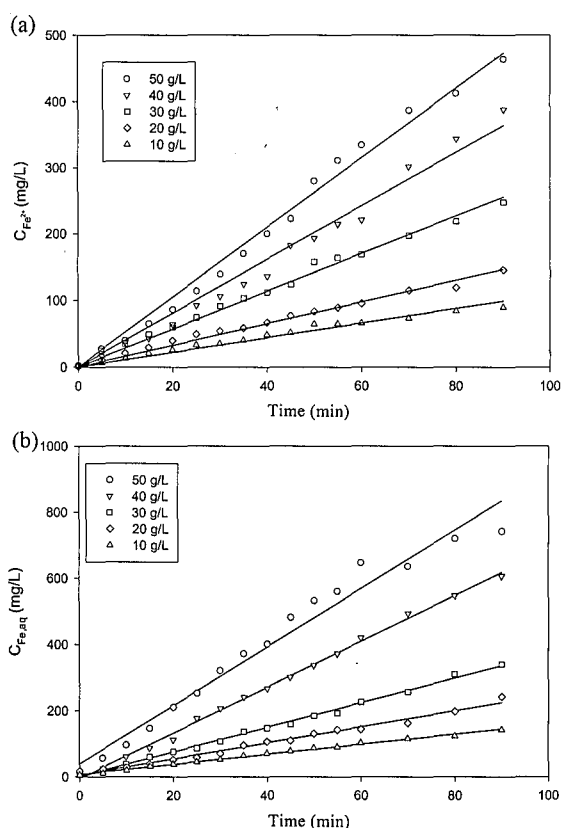


Fig. 2. Time variation of leached (a) Fe^{2+} concentration ($C_{Fe^{2+}}$); (b) total iron concentration ($C_{Fe, aq}$) at various BOF slag loadings. Experimental conditions: pH of solution was 2. \circ , ∇ , \square , \diamond , \triangle : BOF slag concentrations = 50, 40, 30, 20, 10 g/L. The R^2 values of regression lines of \circ , ∇ , \square , \diamond , \triangle : (a) 0.991, 0.988, 0.995, 0.993, 0.982; (b) 0.970, 0.997, 0.996, 0.985, 0.995.

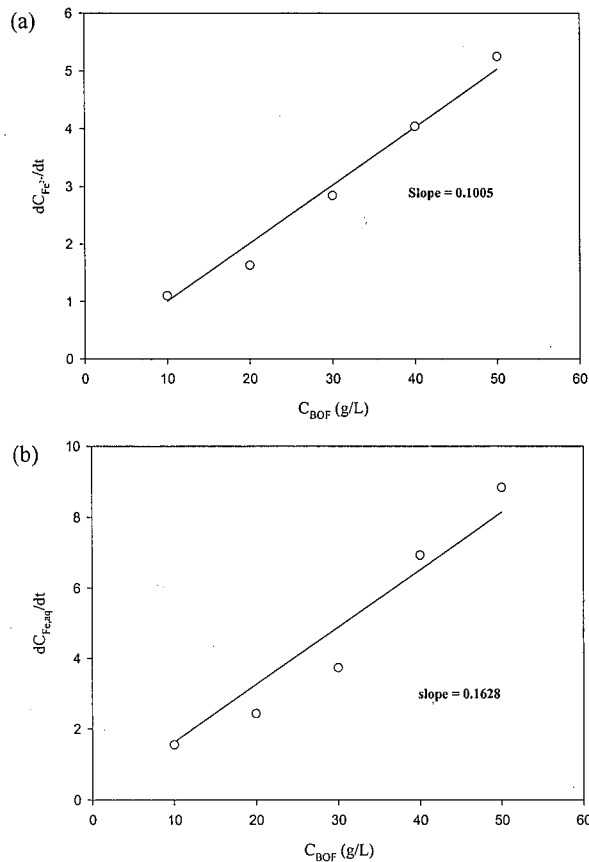


Fig. 3. Relationship between (a) $dC_{Fe^{2+}}/dt$ and C_{BOF} ; (b) $dC_{Fe, aq}/dt$ and C_{BOF} . Experimental conditions are the same as specified in Fig. 2. The R^2 values of the regression lines of (a), (b): 0.974, 0.929.

linearly proportional to reaction time. In the presence of hydrogen peroxide (condition B), ferrous ion reacted with hydrogen peroxide to form "Fenton's reagents" and to produce ferric ion and hydroxyl radical as described in Eq. (1). The hydroxyl radical would further consumed the ferrous ion via Eq. (3). The reactions made the $C_{Fe^{2+}}$ of condition B with H_2O_2 significance lower comparing with the solution absence of hydrogen peroxide (condition A). In the condition C, hydroxyl radical acted as an oxidizing agent to oxidize 2-NS via Eq. (7) in addition to the vigorous reaction between hydroxyl radical and ferrous ion via Eq. (3). Thus, the $C_{Fe^{2+}}$ of condition C was between those of conditions A and B. The production of ferrous ion displayed a non-linear growth in conditions B and C.

pH effects on mineralization of 2-NS

The dissolution of BOF slag to produce ferrous ion is significant only in an acid solution. Also, the action of Fenton's reactions on the degradation of pollutants is more effective under acidic conditions than neutral conditions. As shown in Fig. 5, the mineralization efficiency of TOC_{2-NS} ($\eta_{TOC, NS}$) by the use

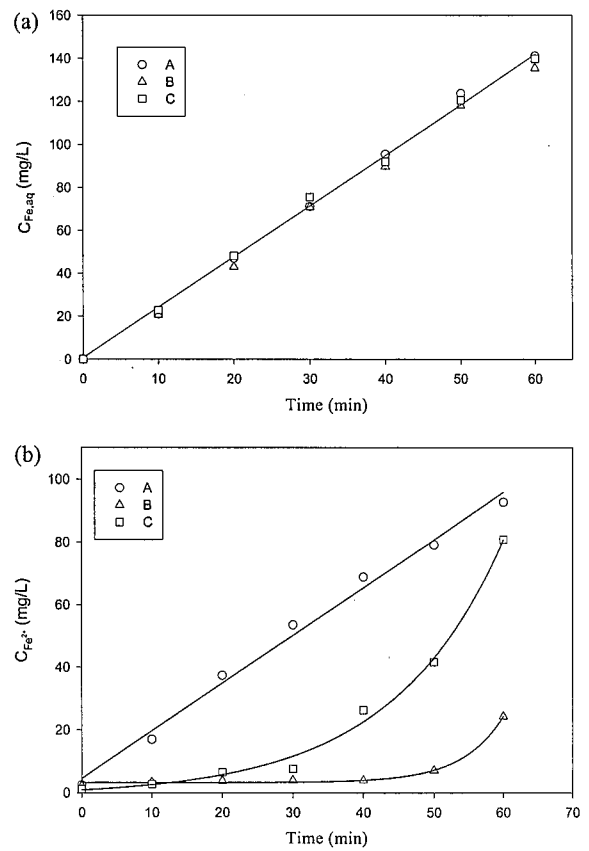


Fig. 4. Time variation of leached (a) total iron concentrations and (b) Fe^{2+} concentration for different conditions. Experimental conditions: A, \circ : 10 g/L BOF slag; B, \triangle : 10 g/L BOF, 350 mg/L H_2O_2 ; C, \square : 10 g/L BOF slag, 350 mg/L H_2O_2 , 100 mg/L 2-NS. The pH value of solution was adjusted by HNO_3 to maintain at 2.

of 10 g/L BOF slag with 350 mg/L H_2O_2 increased with acidity (decrease of pH). Under the experimental conditions, the values of $\eta_{TOC, NS}$ were about 64.7 and 56.3% for pH 2 and 3 after 90-minute reaction time, respectively. However, the $\eta_{TOC, NS}$ was dramatically reduced to 18.9% at a pH value of 4 and to only 3.5% at a pH value of 5. Thus, the better pH values for the effective mineralization of 2-NS were found to be below 3. In order to achieve the better treatment efficiency, the pH value of solution was maintained at 2 for the latter experiments.

Effects of initial H_2O_2 concentration on mineralization of 2-NS

The effect of initial concentration of H_2O_2 (C_{0, H_2O_2}) in the range of 180-1750 mg/L on the $\eta_{TOC, NS}$ of 2-NS was studied at 25°C for 100 mg/L 2-NS and 10 g/L BOF slag. As shown in Fig. 6, the $\eta_{TOC, NS}$ of 2-NS increased with increase in C_{0, H_2O_2} from 180 to 700 mg/L but decreased with further increase in C_{0, H_2O_2} higher than 700 mg/L. Such changes were due to the effects of the ratio of Fe^{2+}

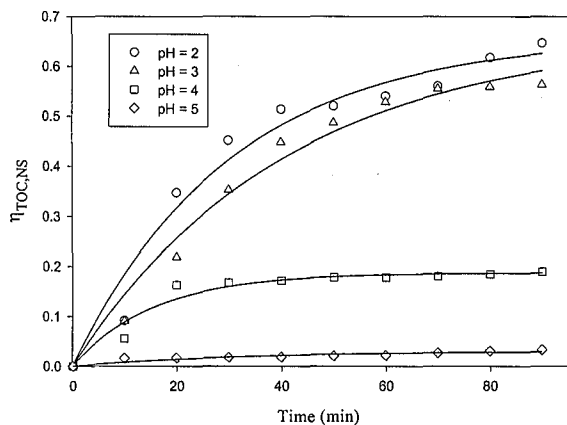


Fig. 5. pH effects on the mineralization of 2-NS ($\eta_{\text{TOC,NS}}$). Experimental conditions: 100 mg/L 2-NS, 350 mg/L H_2O_2 , 10 g/L BOF slag, 25°C. The pH of solution was adjusted by HNO_3 . The R^2 values of regression lines: 0.826 (\circ , pH 2), 0.947 (\triangle , pH 3), 0.982 (\square , pH 4), 0.967 (\diamond , pH 5).

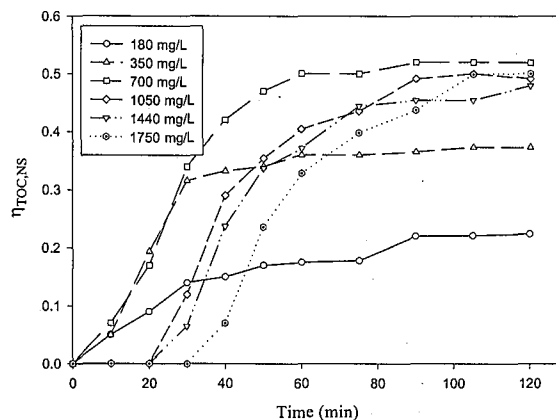


Fig. 6. Effects of initial H_2O_2 concentration on the mineralization of 2-NS ($\eta_{\text{TOC,NS}}$). Experimental conditions: 100 mg/L 2-NS, 10 g/L BOF slag, 25°C, pH = 2.

and H_2O_2 . Equation (1) showed that the stoichiometric molar ratio between the depletion of Fe^{2+} and H_2O_2 is 1. Both Fe^{2+} and H_2O_2 concentrations predominate the reaction. At higher H_2O_2 and Fe^{2+} concentrations, the $\text{OH}\cdot$ production would be increased to act as the sources in the $\text{OH}\cdot$ reactions. An adequate increase in $\text{OH}\cdot$ production should correspond to an increase in the mineralization of 2-NS. However, the reaction rate constant between H_2O_2 and $\text{OH}\cdot$ is as high as $2.7 \times 10^7 \text{ M}^{-1}\cdot\text{s}^{-1}$ (as shown in Eq. (6)). The reaction becomes a predominate reaction in comparison with the reaction between $\text{OH}\cdot$ and 2-NS at further high H_2O_2 concentration, resulting in the reaction of $\text{OH}\cdot$ with H_2O_2 instead of 2-NS. Thus, an increase in concentration of scavenger, such as H_2O_2 and the reaction by-product SO_4^{2-} (from the degradation of 2-NS by Fenton's reaction), will result in an increase in the rate of radical scavenging leading to a reduction in mineralization efficiency. The predomination reaction of $\text{OH}\cdot$ with H_2O_2 also makes the mineralization of 2-NS slow in the early 20-minute reaction time for $C_{0,\text{H}_2\text{O}_2}$ higher than 700 mg/L.

The H_2O_2 consumptions and the Fe^{2+} productions as a function of time at various $C_{0,\text{H}_2\text{O}_2}$ are shown in Figs. 7(a) and 7(b), respectively. The H_2O_2 concentration was nearly zero after 30-minute reaction time, resulting in a gradual reduction of the effects of Fenton's reaction and a quick increase of the concentration of Fe^{2+} in the solution for the cases with $C_{0,\text{H}_2\text{O}_2}$ of 180 and 350 mg/L. Being the Fenton's reaction, most of the Fe^{2+} produced from the dissociation of BOF slag reacted with H_2O_2 to make the free Fe^{2+} presented in the solution in low concentra-

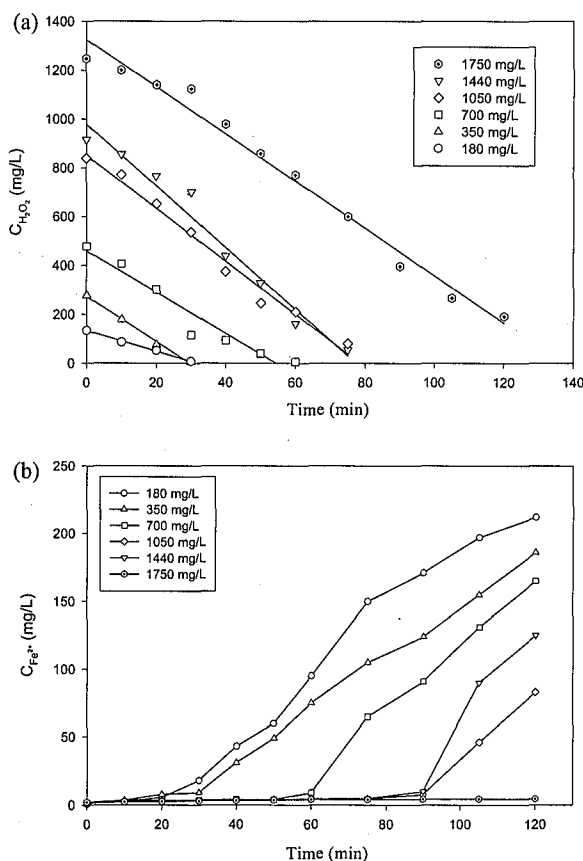


Fig. 7. Time variation of (a) H_2O_2 consumption and (b) Fe^{2+} production for various initial concentrations of H_2O_2 . Experimental conditions are as specified in Fig. 6.

tions for the cases with $C_{0,\text{H}_2\text{O}_2}$ higher than 700 mg/L. Overall, the extent of mineralization of 2-NS increased with increased H_2O_2 concentration, but it was less efficient as $C_{0,\text{H}_2\text{O}_2}$ was higher than 700 mg/L.

Effects of BOF slag loading on mineralization of 2-NS

The effect of BOF slag loading in the range of 3-20 g/L on the mineralization of 2-NS was studied at 25°C for 100 mg/L 2-NS and 350 mg/L H₂O₂. As shown in Fig. 8, a larger BOF slag loading up to 15 g/L resulted in a higher mineralization extent of 2-NS, primarily due to the presence of higher C_{Fe²⁺} with increasing BOF slag loading. A higher C_{Fe²⁺} would produce higher hydroxyl radicals available for the mineralization of 2-NS and would also enhance the utilization of hydrogen peroxide. Noting the complete depletion of H₂O₂ after 30-minute reaction time, the mineralization was stopped for the case with BOF slag loading of 20 g/L (Fig. 9). Further, as shown in Fig. 9, 350 mg/L of H₂O₂ was nearly consumed and free Fe²⁺ was increased rapidly after 30-minute reaction time for the cases with BOF slag loading higher than 10 g/L. The trend of the effects of BOF slag loading on the mineralization of 2-NS was similar to that of the initial H₂O₂ concentration. As the BOF slag loading increased, a relative increase of H₂O₂ concentration was necessary.

Effects of initial concentration of 2-NS on mineralization of 2-NS

Figure 10 shows the variation of mineralization of 2-NS by the use of 10 g/L BOF slag and 350 mg/L hydrogen peroxide at 25°C with various initial concentrations of 2-NS (C_{NS0}) in the range of 30-200 mg/L. It was obvious that a lower C_{NS0} had a higher 2-NS mineralization efficiency. However, as more H₂O₂ was consumed, the increasing rate of mineralization efficiency decreased gradually after 50-minute reaction time.

CONCLUSION

According to the above results and discussion, we have the following conclusions.

- (1) A low pH value can enhance the leaching rate of Fe²⁺ from the basic oxygen furnace slag (BOF slag).
- (2) The mineralization efficiency ($\eta_{\text{TOC,NS}}$) of 2-naphthalensulfonate (2-NS) in terms of total organic carbon (TOC_{2-NS}) by using 10 g/L BOF slag with 350 mg/L H₂O₂ increased with acidity. The adequate pH values for better mineralization of 2-NS were found to be below 3.
- (3) The effect of initial concentration of H₂O₂ (C_{0,H₂O₂}) in the range of 180-1750 mg/L on the mineralization of 2-NS was studied at 25°C for 100 mg/L 2-NS and 10 g/L BOF slag. The extent of mineralization of 2-NS increased with increased

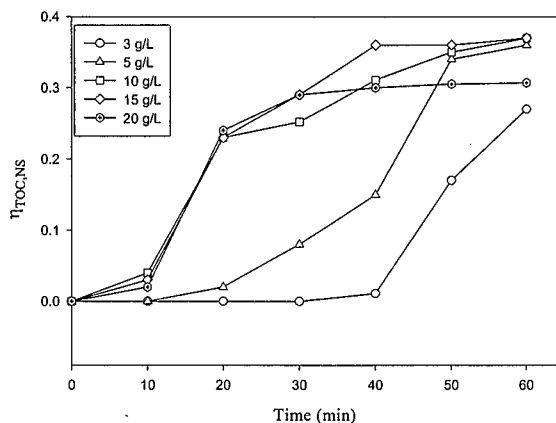


Fig. 8. Effects of BOF loading on the mineralization of 2-NS ($\eta_{\text{TOC,NS}}$). Experimental conditions: 100 mg/L 2-NS, pH = 2, 350 mg/L H₂O₂, 25°C.

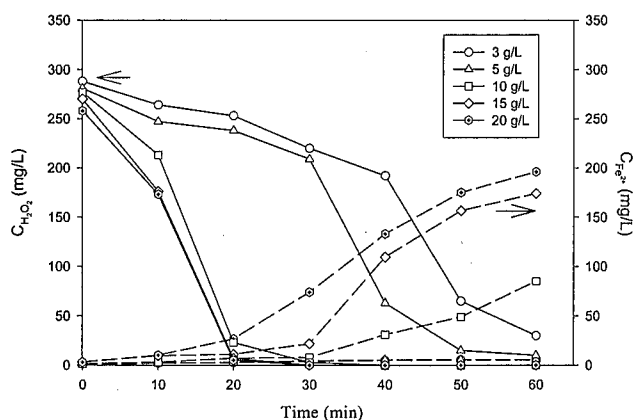


Fig. 9. H₂O₂ consumption and Fe²⁺ production for various BOF slag loadings. Experimental conditions are those as specified in Fig. 8. The dash lines represent C_{Fe²⁺}.

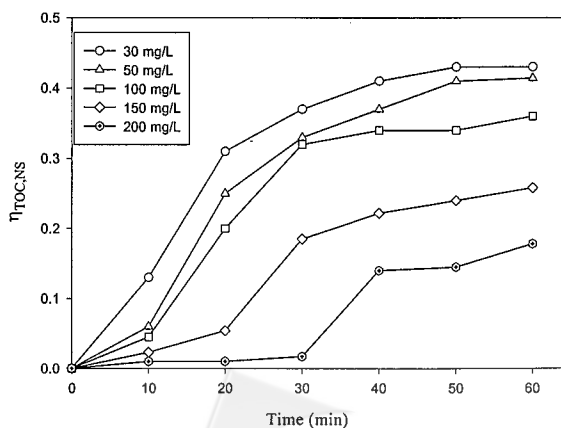


Fig. 10. Time variation of mineralization of 2-NS ($\eta_{\text{TOC,NS}}$) at various initial concentrations of 2-NS. Experimental conditions: pH = 2, 10 g/L BOF slag, 350 mg/L H₂O₂, 25°C.

of mineralization of 2-NS increased with increased C_{0,H_2O_2} , but it was less efficient as C_{0,H_2O_2} was higher than 700 mg/L.

- (4) The higher mineralization extent of 2-NS with increasing BOF slag loading up to 15 g/L was primarily due to the presence of higher Fe^{2+} in the solution with increasing BOF slag loading.
- (5) A lower initial concentration of 2-NS had higher 2-NS mineralization efficiency.

NOMENCLATURE

C_{0,H_2O_2}	initial concentration of H_2O_2 , mg/L
C_{BOF}	amount of BOF slag in aqueous solution, g/L
$C_{Fe^{2+}}$	concentration of ferrous ion in aqueous solution, mg/L
$C_{Fe,aq}$	concentration of total iron ion in aqueous solution, mg/L
C_{NS0}	initial concentration of 2-NS in aqueous solution, mg/L
k_{Fe}^0	leaching rate constant of total iron from BOF slag, mg-Fe/(g-BOF·min)

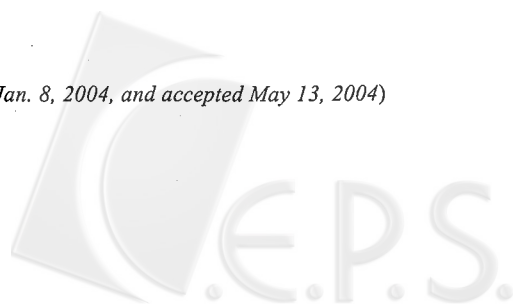
Greek symbol

$\eta_{TOC,NS}$	mineralization efficiency of TOC_{2-NS} , $(TOC_{NS0} - TOC_{NS})/TOC_{NS0}$.
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利用轉爐石催化過氧化氫以分解水中 2-Naphthalenesulfonate

邱求三 張瓊芬 張慶源
國立台灣大學環境工程學研究所

張章堂 李元陞
國立宜蘭大學環境工程學系

摘 要

轉爐石 (BOF slag) 為煉鋼廠於煉鋼過程中所產生之廢棄物，其含有大量的 FeO 及 Fe₂O₃，可嘗試將其作為分解 H₂O₂ 以啟動費頓反應的催化劑；2-Naphthalenesulfonate (2-NS) 為紡織工業及印刷電路板工業廢水中之有機污染物，其易溶於水但不易被傳統廢水之生物處理方法予以分解，所以 2-NS 的分解便需利用高級氧化程序；本研究嘗試利用轉爐石中的鐵來催化 H₂O₂ 以產生氫氧自由基，再利用氫氧自由基的高氧化性來達到分解 2-NS 的目的，實驗結果顯示，溶液 pH 值小於 3，較有利於 2-NS 之礦化，當轉爐石及 H₂O₂ 添加量分別為 15 g/L 及 700 mg/L 時，可得到較佳之 2-NS 礦化效率。

