

Microwave-induced Degradation of Chlorobenzene Using Fe⁰ and TiO₂ Coated on Cordierites as Catalyst

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Abstract

In this research, MW is used to irradiate and induce Fe⁰/cordierite (Fe⁰ coated cordierite particles) and TiO₂/cordierite (TiO₂ coated cordierite particles) for improving the catalytic decomposition rate to remove chlorobenzene (CB) dissolved in aqueous solution. Laboratory results show that when 30 W of microwave energy is applied for 300 sec the irradiation of microwave than without microwave irradiation improves the CB removal efficiency by 3.2 times (57.4% vs. 18.2%) for Fe⁰/cordierite and 2.7 times (43.3% vs. 15.8%) for TiO₂/cordierite. Hence, applying the microwave induced cordierite coated Fe⁰ or TiO₂ particles is a new application that has been proved effective to remove chlorine-containing organic pollutants.

Keywords: microwave, Fe⁰/cordierite, TiO₂, TiO₂/cordierite

1. Introduction

Chlorine-containing organic pollutants may be treated using thermal treatment, biological degradation, advanced chemical oxidation, and incineration among the many others. However, each of the above methods may have one or several problems or difficulties. For example, the thermal adsorption method is used to recover the organic pollutant, but the high temperature will usually destruct the pollutant to be recovered (Ai, Yang, & Lu, 2005; Lien & Zhang, 2005; Takashima, Karches, & Kanno, 2008); the biological degradation is possible only when the microbial growth conditions are well maintained in addition to relatively long reaction time (Janda, Vasek, Bizova, & Belohlav, 2004; Lin, Lo, & Liou, 2004; 2005); the advanced chemical oxidation is expensive and is not effective in removing organic matter that may not be easily oxidized or have relatively high chlorine content (Liu et al., 2001); the incineration method may produce intermediate products that may be even more refractive or toxic than the original chlorine-containing organic pollutants (Lien & Zhang, 2002; Zhong, Yang, Ju, & Cheng, 2009).

Using nano-scale zero-valent iron as electron sources has been proved effective in removing chlorine-containing organic pollutants from wastewater and groundwater in recent years (Lin et al., 2004; Oh, Chiu, Kim, & Cha, 2006). However, zero-valent iron has several disadvantages such as the difficulty to maintain its surface activity, the formation of a layer of oxidized iron on the surface within a short reaction period, and the difference between surface activities for zero-valent irons from various sources (Lin et al., 2004; Ruiz, Seal, & Reinhart, 2000; Lin & Lo, 2005; Lee & Jou, 2012). On the other hand, TiO₂ has better photochemical activities; it is more stable in aqueous solution, less expensive, lower toxicity to adversely impact the environment, and better removal efficiency (Jou & Wu, 2008; Yamazaki et al., 2001; Ai, Yang, & Lu, 2005). Some disadvantages involved in the use of TiO₂ to treat VOCs include the production of phosgene, carbon monoxide, and toxic byproduct such as carbon tetrachloride (Mohseni, 2005). Further, this method applies higher energy to decompose the pollutant so that its application is limited because of tremendous energy requirement (Ai, Yang, & Lu, 2005).

Microwave (MW) is an electro-magnetic radiation with frequencies ranging from 300 MHz to 300 GHz (Chang et al., 2012; Woo, Grippin, Wu, & Wander, 2012). The MW absorbed by a solution will cause non-thermal effect to reduce the activation energy and weaken the chemical bonds of the organic chemicals (Zhang et al., 2007).

Successful application of MW technology depends on selecting appropriate media for treating organic matter including microwave combined with granular activated carbon or zero-valent iron nano-particles for treating pentachlorophenol (Jou, 2008; Jou & Wu, 2008; H. Lee, C. Lee, & Jou, 2010a) and improving the efficiency of TiO_2 photocatalyst (Jou, Lee, Tsai, & Wang, 2008) to save energy while improving efficiencies (Liu, Quan, Bo, Chen, & Zhao, 2004). Lee et al. (2009b; 2010b) and Jou et al. (2010a; 2010b) applied MW radiation induced zero-valent iron nano-particles for enhancing the CB decomposition; the results show that when MW radiation penetrates the solution to reach the surface of suspended Fe^0 particles, the radiation accelerates the iron oxidization, and increases the surface activity site thus enhancing the CB decomposition rate (Lee, Jou, & Huang, 2009b; Lee, Jou, & Wang, 2010b; Lee & Jou, 2012; Jou, Lee, Lin, & Huang, 2010a; Jou, Wu, & Lee, 2010b).

In this research, a new method of treating chlorine-containing organic pollutants with an effective catalyst that combines the strong reductive capability of Fe^0 and the TiO_2 photo-catalytic characteristics TiO_2 with superior thermal conductivity and chemical stability of cordierite (i.e. Fe^0 /cordierite and TiO_2 /cordierite, respectively) irradiated with low MW energy (30 MW) for enhancing chlorobenzene removal is studied.

2. Materials and Methods

2.1 Reagent

The chlorobenzene solution was prepared by dissolving 905 μL of chlorobenzene (99.9% purity GR Reagent, TEDIA, USA) in methanol (99.9% purity, GR Reagent, TEDIA, USA) with the final concentration of the stock solution adjusted to 20000 mg L^{-1} . When needed, 250 μL of the stock solution was dissolved in de-ionized water (18.2 M Ω , Millipore Co, USA) with the final volume adjusted to yield 100 mg L^{-1} chlorobenzene.

2.2 Coating Cordierite with Zero-valent Iron and TiO_2

For preparing Fe^0 /cordierite, cordierite particles were washed in 37% boiling HCl solution for 1.5 hours to increase its surface area. The acid-washed cordierite was suspended in de-ionized water to be cleaned using ultrasonic agitation and then boiled for 30 minutes. These cleaning procedures were repeated several times until the cordierite became neutral. After being dried in 105 $^{\circ}\text{C}$ oven for 24 hours, the cordierite particles were suspended in $\text{FeCl}_3 \cdot \text{H}_2\text{O}$ solution and the suspension was agitated (200 rpm) in a 25 $^{\circ}\text{C}$ water bath for 4 hours. Afterward, the cordierite particles were removed from the FeCl_3 solution; they were added with NaBH_4 solution dropwise until all cordierite particles were submerged in NaBH_4 solution. The un-adsorbed FeCl_3 was removed from cordierite particles by washing with de-ionized water. Based on weight, the final dried Fe^0 /cordierite particles are covered with 100 mg zero-valent iron per gram of cordierite.

For preparing TiO_2 /cordierite, 25 mL $\text{Ti}(\text{OC}_3\text{H}_7)_4$ was mixed with 25 mL $\text{CH}_3(\text{CH}_2)_2\text{OH}$ and 20 mL CH_3COOH ; the mixture was agitate at 800 rpm for 8 hours. When precipitates were observed, cordierite particles were added to the mixture. The mixture was further mixed at 800 rpm for 8 hours. Afterward, the mixture was allowed to form semi-transparent gel under quiescent conditions. After dried at 150 $^{\circ}\text{C}$ followed by calcined at 550 $^{\circ}\text{C}$ for 2 hours, the final dried product is covered with 118 mg TiO_2 per gram of cordierite.

2.3 Experimental Methods

The background information on CB adsorption was obtained by placing 1 g cordierite and 1 g Fe^0 /cordierite in 40 mL of 100 mg L^{-1} CB solution contained in a set of brown bottles that can be sealed with Teflon-lined screwed caps. The bottles were placed in 25 $^{\circ}\text{C}$ constant-temperature water bath, and the contents were removed after 30, 60, 90, 120, 150, 180, 210, and 240 minutes for analyses. For testing the efficiency of CB removal with MW treatment, the intermittent MW irradiation study was carried by placing 1 g cordierite, 1 g Fe^0 /cordierite and 1 g TiO_2 /cordierite, respectively, in 50 mL of 100 mg L^{-1} CB contained in boro-silica glass reactors. The reactors were then placed in MW oven to be irradiated intermittently with 30W MW for 15 cycles: each cycle contains 20 seconds of MW irradiation followed by 120 seconds of no MW irradiation. The total irradiation time was 300 seconds.

2.4 Analyses

Chemical analyses of organic intermediates and final products matter was performed with HP 6890 gas chromatography (GC) using the HP-5MS capillary column; the chromatography is equipped with an HP 5973 mass selective detector (MSD). The constant carrying gas (He) flow rate was maintained at 1.5 mL min^{-1} . The oven temperature was programmed from 70 to 260 $^{\circ}\text{C}$ (hold for 5 min) at a ramp rate of 30 $^{\circ}\text{C min}^{-1}$. The GC injector temperature was 250 $^{\circ}\text{C}$ and MSD injector temperature was 320 $^{\circ}\text{C}$.

3. Results and Discussion

3.1 CB adsorption on Cordierite

Under 25 °C, the adsorption of CB contained in 40 mL of 100 mg L⁻¹ CB by 1 g cordierite reaches saturation at 240 minutes with 5.33 mg CB adsorbed. The CB adsorption data are fitted with the Langmuir and the Freundlich isotherms by plotting the quantity of CB absorbed per unit quantity of cordierite (C_e/Q_e) vs. the equilibrium CB concentration in the solution (C_e). As shown in Figure 1, the Langmuir isotherm equation is $y = 81.701x - 7890$ with R^2 value of 0.5989 and adsorption constant of 0.0104. Plotting the logarithmic values of Q_e ($\ln Q_e$) vs. the logarithmic value of C_e ($\ln C_e$) in Figure 2 leads to the Freundlich isotherm of $y = -69.024x + 316.81$ with R^2 of 0.8983, and adsorption constant of 0.0145. Based on the value of R^2 , the Freundlich isotherm fits the experimental adsorption data better than the Langmuir isotherm. This is because that the latter assumes a homogeneous surface with no inter-molecular attraction among the solute to be adsorbed whereas the Freundlich isotherm assumed heterogeneous adsorbent surface that is more realistic to account for the adsorption of CB on cordierite surface.

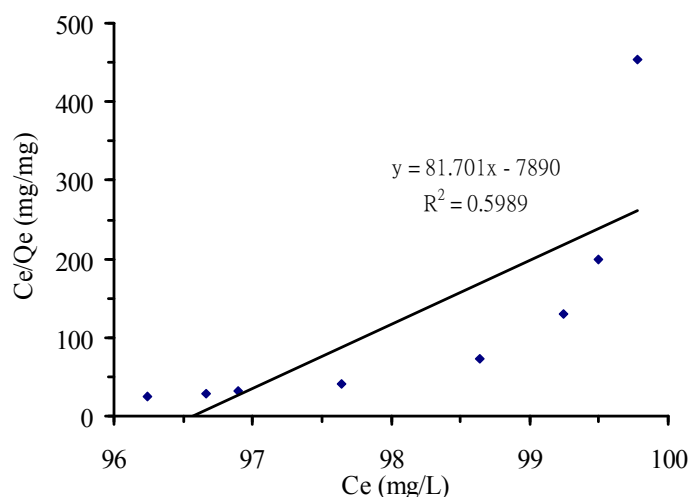


Figure 1. The Langmuir isotherm for the adsorption of CB on cordierite surface

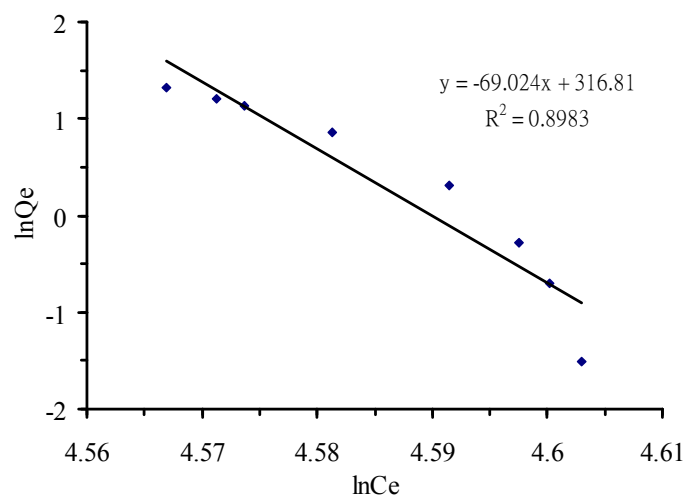


Figure 2. The Freundlich isotherm for the adsorption of CB on cordierite surface

3.2 Influence of Coating Cordierite Surface with Zero-valent Iron and TiO₂ on CB Removal

When Fe⁰/cordierite particles contact CB solution, CB is diffused and adsorbed on cordierite surface where it oxidizes zero-valent iron directly to result in electron transfer (Xiong, He, & Zhang, 2000) so that the adsorbed CB is de-chlorinated (Chena, Al-Abed, Ryanb, & Li, 2001). Figure 3 shows that after 240 minutes, 18.2% CB is

removed with $8.0 \times 10^{-4} \text{ min}^{-1}$ reaction rate constant.

In contrast, the TiO_2 /cordierite particle surface absorbs the MW energy to produce the electron-electronic hole pair that oxidizes and decomposes the adsorbed CB molecules. Under conditions of complete darkness by wrapping the amber reactor with aluminum foil, and 25°C for 240 minutes, the CB removal rate increases with reaction temperature (Figure 3) and the CB removal efficiency reaches 15.8% with $7.0 \times 10^{-4} \text{ min}^{-1}$ reaction rate constant.

Under the same experimental conditions (i.e. 25°C and 240 minute reaction time), the CB removal rate is 18.2% for Fe^0 /Cordierite particles that is greater than the 15.8% for TiO_2 /cordierite particles. If the maximum quantity of 5.33 mg CB being absorbed by plain cordierite is deducted, the net CB removal efficiencies are 12.7% are for Fe^0 /cordierite and 10.4% for TiO_2 /cordierite. The Fe^0 coated cordierite has a better CB removal efficiency than TiO_2 coated cordierite because zero-valent iron has a higher activity so that more electrons can be easily released to favor the CB decomposition reactions. Additionally, the formation of electron-electron hole pairs by TiO_2 must be excited and catalyzed by an external light source. In total darkness, the efficiency of CB decomposition by TiO_2 /cordierite is greatly impaired.

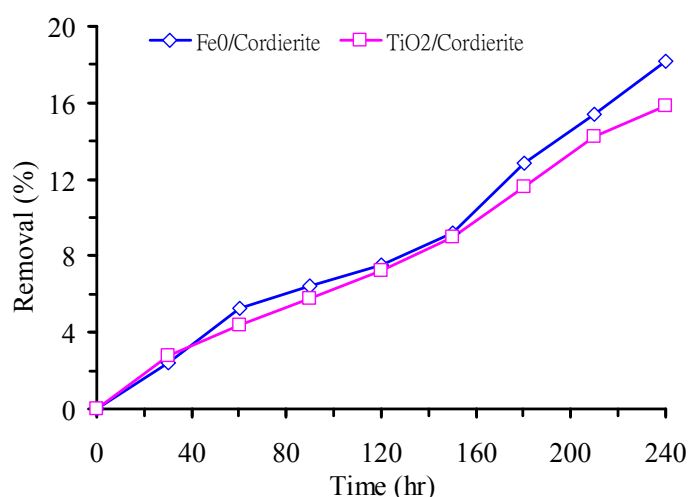


Figure 3. Comparison of CB removal efficiencies for Fe^0 /cordierite and TiO_2 /cordierite

3.3 CB Removal Efficiency for Plain Cordierite with MW Irradiation

When a medium is placed in an electrical or electromagnetic field, its molecules become polarized to rotate and move vigorously leading to the production of friction that will raise the heat. The polarization causes the molecules to be at a higher energy state that brings about more impacts among the reactant molecules, and faster decomposition of pollutants occur (Zhang et al., 2007; Chang et al., 2012). The MW heating is based on using media with higher dielectric loss that is related to its own dipole orientation. In this study, intermittent MW irradiation applied on 1 g cordierite suspended in 50 mL of 100 mg L^{-1} CB solution (Figure 4) causes the CB to decompose by 14.8 mg. Under similar experimental conditions, the MW irradiation on CB solution without cordierite particles will cause the CB to drop only by 8.3 mg. Additional, without MW irradiation, cordierite will adsorb the maximum amount of 5.33 mg CB. These results indicate that the irradiation of cordierite with 30 W MW for 300 seconds is not effective in removing CB cordierite suspended in 50 mL of 100 mg L^{-1} CB solution.

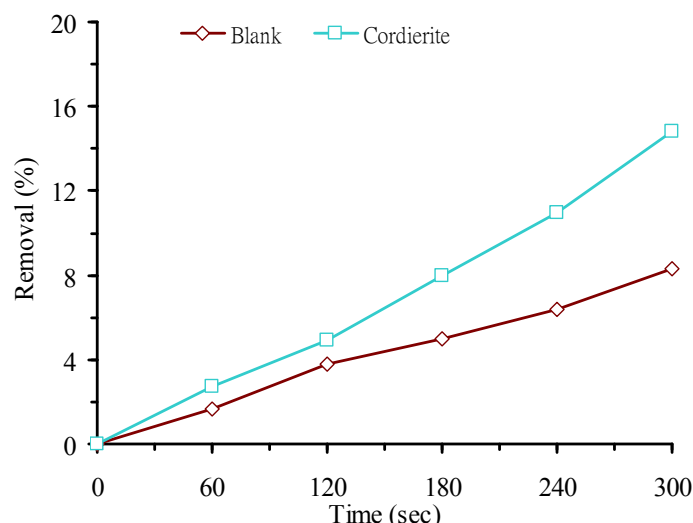


Figure 4. Removal of CB vs. MW irradiation time for 1 g

3.4 Influence of MW Irradiation on CB Removal for Cordierite Coated With Fe^0 and TiO_2

Zero-valent iron serves as an electron provider when involved in the reductive de-chlorination reactions. Under normal temperature, it has high oxidation characteristics to become oxidized forming Fe^{+2} rapidly; the self-oxidation process releases heat so that the metal oxidation rate is further enhanced. Relative longer MW irradiation with more MW absorbed by Fe^0 /cordierite confirms that the MW energy is effective in inducing the capability of cordierite to decompose CB from aqueous solution as seen by less reaction time with more removal efficiency. When irradiated with 30 W MW output energy intermittently for 300 seconds in 15 cycles consisting of 20-second irradiation period followed by 120 seconds of no irradiation, the results shown in Figure 5 indicates that 57.4% CB can be removed from 80 mL aqueous solution containing 100 mg L^{-1} CB by 1 g Fe^0 /cordierite.

Under similar intermittent MW irradiation conditions, Figure 5 also indicates that 43.3% of CB can be removed by 1 g TiO_2 /cordierite from 80 mL of 100 mg L^{-1} CB solution. Major forms of crystalline structure contained in TiO_2 are anatase and rutile; the gap between valence band and conductance band is greater for anatase that leads to longer electron-holes life, faster electron transfer rate and lower recombination of electron-hole pairs. Hence, when TiO_2 oxides are excited by MW irradiation, electrons in the valence band are elevated to the conductance band to form electron-hole pairs. In addition to oxidizing or reducing the adsorbed compounds, these pairs will also inhibit the electron and hole recombination, prolong the photo-catalytic reaction period and provide more paths for photodecomposition of organic pollutants (Dindar & İçli, 2001). If only water molecules are irradiated by MW, the $\cdot\text{OH}$ free radicals will not be produced. When powder TiO_2 is added, the MW irradiation will excite TiO_2 to produce electron-hole pairs. Under appropriate conditions, $\cdot\text{OH}$ free radicals are formed to promote the oxidative decomposition of organic compounds (Horikoshi & Serpone, 2009; Zhang et al., 2007). Horihoshi et al. (2009) also observed that combining MW radiation with photo-catalyst based rotations of electronic resonance will produce 20% more $\cdot\text{OH}$ free radicals than photo-catalyst alone.

The mechanisms for decomposing CB dissolved in aqueous solution by Fe^0 /cordierite and TiO_2 /cordierite are different. For Fe^0 /cordierite, the direct oxidation of zero-valent iron into ferric ions through electron transfer by intermediates at Fe^0 surface results in the reductive decomposition of chlorine-containing organic matter (Xiong et al., 2000). For TiO_2 /cordierite, valence electrons elevated to conduction band causes the formation of electron-electronic holes that will react with the adsorbed CB molecules. When irradiated with MW, the electron-hole pair will enhance the metal catalyst surface oxidation rate, increase the surface activation site, speed up the CB degradation rate, and inhibit the electron and hole recombination so that the photo-catalytic reaction time is prolonged to decompose more pollutants with better removal efficiency.

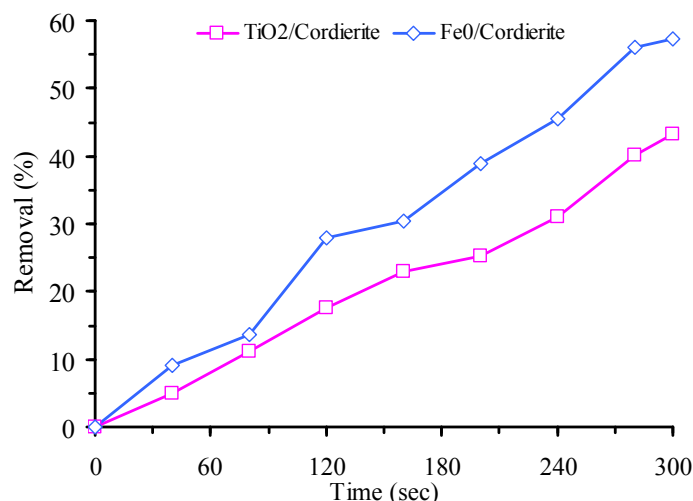


Figure 5. MW removal vs. MW irradiation time for Fe⁰/cordierite and TiO₂/cordierite

4. Conclusions

The use of MW to irradiate Fe⁰/cordierite (Fe⁰ covered cordierite particles) and TiO₂/cordierite (TiO₂ covered cordierite particles) in order to raise the efficiency of removing CB from aqueous solution has been studied. Under MW irradiation, the CB removal efficiencies are raised 3.2 times (57.4% vs. 18.2%) for Fe⁰/Cordierite and 2.7 times (43.3% vs. 15.8%) for TiO₂/cordierite. For Fe⁰/cordierite, the MW irradiation enhances the oxidation rate of metal catalytic on cordierite surface, raise the surface activity to provide more contact between the catalyst and CB molecules so that the decomposition reaction rate of the organic chlorine-contained pollutant is enhanced. For TiO₂/cordierite, the MW irradiation raises the TiO₂ valent electrons to conduction band to form electron-electronic holes that reduce the pollutant molecules adsorbed on cordierite surface, inhibit the recombination of electrons and holes. This leads to prolonged photo-catalytic time to result in more paths for decomposing the pollutant photo-catalytically. Therefore, using the combined MW irradiation with Fe⁰/cordierite or TiO₂/cordierite is an effective and energy-efficient method for treating chlorine-containing organic matter.

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