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## Influence of supports on photocatalytic degradation of phenol and 4-chlorophenol in aqueous suspensions of titanium dioxide

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### Abstract

The photocatalytic degradation of phenol and 4-chlorophenol (4-CP) in aqueous suspensions with the use of titanium dioxide (TiO<sub>2</sub>) under UV irradiation was examined. The effects of different supporting materials mixed physically with TiO<sub>2</sub> were studied to achieve maximum degradation efficiency. Among the three supports, namely activated carbon (AC), silica (SiO<sub>2</sub>) and zeolite (ZSM-5), all exhibited paramount efficiency for degradation of phenol and 4-CP and was better than TiO<sub>2</sub> alone. The optimum concentration was found to be 50 mg for all supporting materials. The efficiency order of the three supports was as follows: AC > ZSM-5 > SiO<sub>2</sub>, respectively. Whilst, the degradation of phenol and 4-CP was improved from 70.6% to 87.6% and 80.6% to 89.7%, respectively, within 120 min photocatalysis in the presence of optimal amount of AC. The degradation was also comparatively enhanced in the presence of cheaper rice husk and the activity was closed to ZSM-5 and lower than AC.

**Key words:** 4-chlorophenol; phenol; photocatalytic degradation; support; TiO<sub>2</sub>

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### Introduction

Water pollution by organic and inorganic contaminants is of immense public concern. A typical group of organic contaminants is the phenols and its derivative such as 4-chlorophenol (4-CP). These are important environmental pollutants because of their toxic effect, ubiquitous presence and carcinogenic character towards life in the aquatic environment (Adav et al., 2007). Phenol and its derivatives are some of the most refractory contaminants, which may occur in the environment due to its widespread use in agriculture, petrochemical, textile, paint, plastic, production processes of pesticides and dyes. These contaminants have led to the priority pollutants by the United States Environmental Protection Agency due to their harmful effect to organisms at low concentration (Calce et al., 2002). These compounds have recently been of great concern because of the acute toxicity, carcinogenicity and/or persistency in the environment (Aksu, 2005). These compounds are transformed or degraded very slowly by natural processes (Vogel et al., 1987), having significant water solubility and have to be eliminated to preserve the environmental quality.

The conventional methods for removing organic contaminants from water such as activated carbon adsorption, microbial degradation, solvent extraction and chemical oxidation are frequently used (Safarik et al., 1997; Slokar and Le Marechal, 1998; Chung et al., 2003; Denizli et al., 2005). The high cost of activated carbon, solvent extraction and oxidation treatments has stimulated interest to use cheaper raw materials. Heterogeneous photocatalysis utilizing titanium dioxide (TiO<sub>2</sub>) is a well known emerging advanced oxidation process for the effective removal of organic contaminants in water and air (Ollis et al., 1991; Hoffmann et al., 1995; Pearl et al., 1997; Wang et al., 2009). TiO<sub>2</sub> is of great interest because of photocatalytically stable, non-toxic nature, low cost material, chemically and biologically inert, easy to produce and to use without risks to environments and humans (Serpone et al., 1995, Akpan and Hameed, 2009; Ma et al., 2011; Li et al., 2011). TiO<sub>2</sub> exhibits high adsorption ability to organic contaminants, corresponding with good quality photocatalytic efficiency. However, shortcoming of using TiO<sub>2</sub> in photocatalytic processes is its rapid aggregation in a suspension resulting in decrease of effective surface area in addition to recombination of generated electron-hole pairs. This disadvantage of TiO<sub>2</sub> results in low catalytic efficiency.

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Various methods are documented to improve photocatalytic efficiency of TiO<sub>2</sub> (Serpone et al., 1995; Yu et al., 2002; Neppolian et al., 2002; Kuo and Lin, 2004; Karunakaran et al., 2010; He et al., 2011; Li et al., 2011). Another effective method that might be able to increase the photocatalytic efficiency of TiO<sub>2</sub> is to add a support or co-sorbent material such as silica (SiO<sub>2</sub>), alumina (Al<sub>2</sub>O<sub>3</sub>), zeolite (ZSM-5) or clay and activated carbon (AC) (Minero et al., 1992; Anderson and Bard, 1995, 1997; Lepore et al., 1996; Xu and Langford, 1997; Torimoto et al., 1997; Xu et al., 1999; Yoneyama and Torimoto, 2000; Shimizu et al., 2002). The dispersion of TiO<sub>2</sub> with supports has become attractive because of supports' adsorption capability which results into composite photocatalyst (Moafi et al., 2011). TiO<sub>2</sub> with supports or co-sorbent offers high specific surface area, which helps in more effective adsorption than TiO<sub>2</sub> alone (Anderson and Bard, 1995, 1997; Xu and Langford, 1995, 1997; Torimoto et al., 1997; Takeda et al., 1997). The synergy between TiO<sub>2</sub> particle and the support enhances the degradation which is attributed to reduction in the electron-hole recombination reaction on the surface (Lopez Nieto, 2001). Over the years, the opportunity of producing silica and activated carbon from cheaper and readily available source like rice husk (RH) for water purification is appealing. Rice is one of the main crops, which is widely cultivated in Asian countries. The RH is an agricultural residue produced in the milling process when the grain is separated from the outer covering (husk). They are the natural sheaths that form on the rice grains during their growth.

Earlier we have reported the photocatalytic degradation of phenolic compounds under the influence of TiO<sub>2</sub> (Naeem and Ouyang, 2009a, 2009b; Naeem et al., 2010) and iron-doped TiO<sub>2</sub> (Naeem and Ouyang, 2010) nanoparticles. In this ongoing study, the influence of the supports such as AC, SiO<sub>2</sub> and zeolite (ZSM-5) on the photocatalytic activity of pure TiO<sub>2</sub> was evaluated using the degradation of phenol and 4-CP. Thus the prime objective of the present work was to improve the competence of photocatalytic process using supports. Results were also compared utilizing cheap material RH as an alternative source of SiO<sub>2</sub> and AC.

## 1 Experimental

### 1.1 Materials

TiO<sub>2</sub> photocatalyst of P25 was the product of Degussa Co., Germany. It is mostly in the anatase form (80% anatase and 20% rutile) with a reactive surface area of (50 ± 15) m<sup>2</sup>/g and an average particle size of 21 nm. Analytical reagent grade phenol and hydrochloric acid were obtained from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). High purity 4-CP was purchased from Aladdin Chemical Reagent Co., China. AC (SSA

950 ± 10 m<sup>2</sup>/g) was obtained from Ningxia Coal Co., Ltd. (China). ZSM-5 (Si/Al = 50, 5-μm, 320 m<sup>2</sup>/g) was obtained from Nankai University (Tianjin) and SiO<sub>2</sub> (125–5 μm, 460 m<sup>2</sup>/g) was purchased from Qingdao Haiyang Chemical Co., Ltd. (China). All chemicals were used as provided by the suppliers without further purification.

The co-sorbent RH was a free gift from a farmer near Chang Chun (China). The RH was crushed and sieved with an 80-mesh sieve. Then the RH was washed with distilled deionized water to remove all dirt and then oven-dried at 80°C till constant weight. The dried RH was stored in a polythene plastic bag and kept in a desiccator, and was used as such without any further physical or chemical treatment. All aqueous solutions were prepared with deionized distilled water obtained by further purification of distilled water with Milli-Q Gradient A-10 system (Millipore Corporation) to a specific resistance better than 18 MΩ·cm.

### 1.2 Photoreactor and light source

As for photocatalytic degradation, irradiation was performed in an open Pyrex-glass cell with 500 mL capacity (of 8.5 cm inside diameter and 15 cm height). The design description of photocatalytic reactor was reported in our earlier study (Naeem and Ouyang, 2009b). Irradiation was carried out using a 125-W bulb from Leijian Special Light Source Co., Ltd. (Shenzhen, China) with a wavelength of 365 nm.

### 1.3 Photocatalytic measurement

A suspension was prepared by adding 50 mg of TiO<sub>2</sub> to a 200 mL of phenol or 4-CP aqueous solution (1 × 10<sup>-4</sup> mol/L) using appropriate amount of support or RH at pH 5. The pH of suspension was measured using PH-035 digital pH meter from Changlilai Technology Co., Ltd. (Shenzhen, China) by adding HCl (0.1 mol/L). All the experiments were performed at an ambient temperature (25 ± 1°C) and atmospheric pressure. During the experiment, air was continuously bubbled into the catalyst suspension. Prior to irradiation, the suspension was magnetically stirred in the dark for at least 15 min to ensure the establishment of an adsorption/desorption equilibrium. Subsequently, the light was turned on and it was treated as the starting point (*t* = 0) of the reaction. Suspensions were continually stirred also during the runs.

At 20 min intervals for 120 min, the samples of 7 mL were retrieved from the reactor and immediately centrifuged at 3000 r/min for 10 min, then filtered through a 0.45-μm Millipore filter to remove the TiO<sub>2</sub> particles. The filtered samples were stored at 4°C prior to analysis. The characteristic absorption intensities of phenol (at 270 nm) and 4-CP (at 278 nm) was measured by using Helios Gamma UV-Vis spectrophotometer to monitor the variation in concentration of phenol and 4-CP during the photocatalytic degradation runs. The degree of degradation (*X*) of each

sample was computed using following equation:

$$X = \frac{A_0 - A_t}{A_0} \quad (1)$$

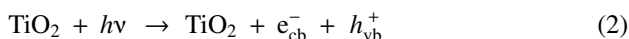
where,  $A_0$  is the absorbance at time zero and  $A_t$  is the absorbance at any subsequent reaction time.

## 2 Results and discussion

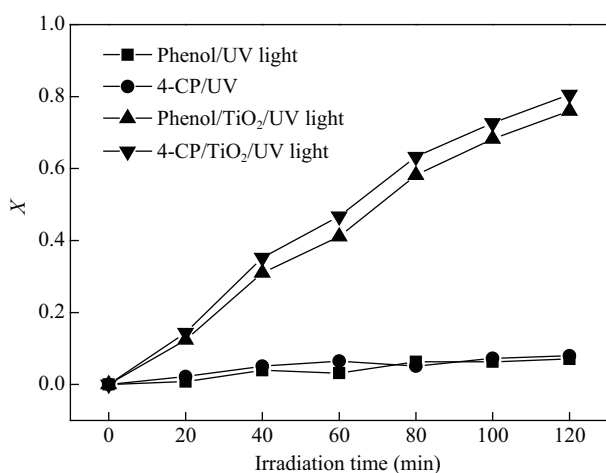
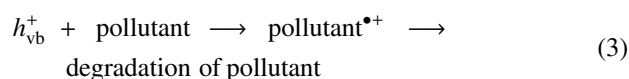
### 2.1 Photocatalytic degradation of phenol and 4-CP

Aqueous solutions of phenol and 4-CP were irradiated in the presence or absence of TiO<sub>2</sub>-P25 by 125 W UV-lamp. The role of photocatalytic degradation and the effect of direct photolysis on the degradation of phenol and 4-CP were studied. **Figure 1** shows the change in degree of degradation versus irradiation time of aqueous solution of phenol and 4-CP. Control experiment was performed by employing UV-irradiated blank solution. The degree of degradations of phenol and 4-CP were negligible when the aqueous solution was irradiated without TiO<sub>2</sub>. Degradation of phenol and 4-CP was only 8% within 120 min in the direct photolysis signifying that the photocatalysed degradation in the presence of TiO<sub>2</sub>/UV is particularly recognized to the photocatalytic reaction of the TiO<sub>2</sub> particles.

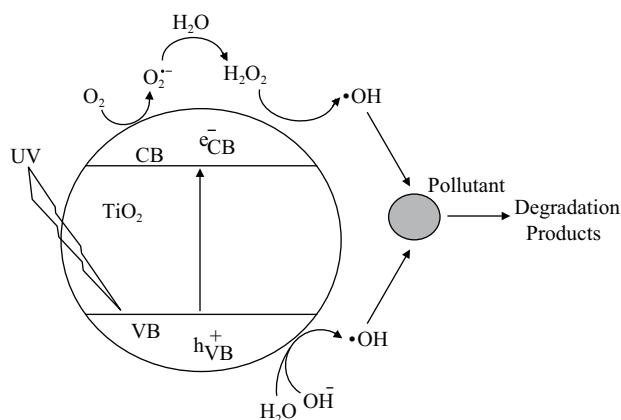
It has been established that photocatalysed degradation of organic pollutant in aqueous solution is initiated by the photoexcitation of the TiO<sub>2</sub>, followed by the formation of an electron-hole pair on the surface of catalyst (Daneshvar et al., 2004):



The high oxidation potential of the hole ( $h_{\text{vb}}^+$ ) allows the oxidation of organic pollutant (phenol and 4-CP) to degradation product.

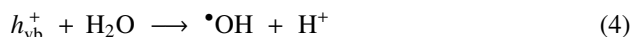


**Fig. 1** Effect of UV light and TiO<sub>2</sub> particles on photodegradation (X) of phenol and 4-CP.



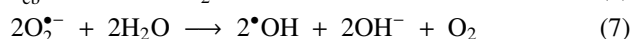
**Fig. 2** General mechanism of TiO<sub>2</sub> under UV light photoexcitation.

Whereas very reactive hydroxyl radicals ( $\bullet\text{OH}$ ) can also be formed either by the decomposition of water or by the reaction of the hole with  $\text{OH}^-$ ,



The  $\bullet\text{OH}$  is an exclusively strong, non-selective oxidant, which conducts the partial or complete mineralization of organic pollutants (Daneshvar et al., 2004; Behnajady et al., 2006).

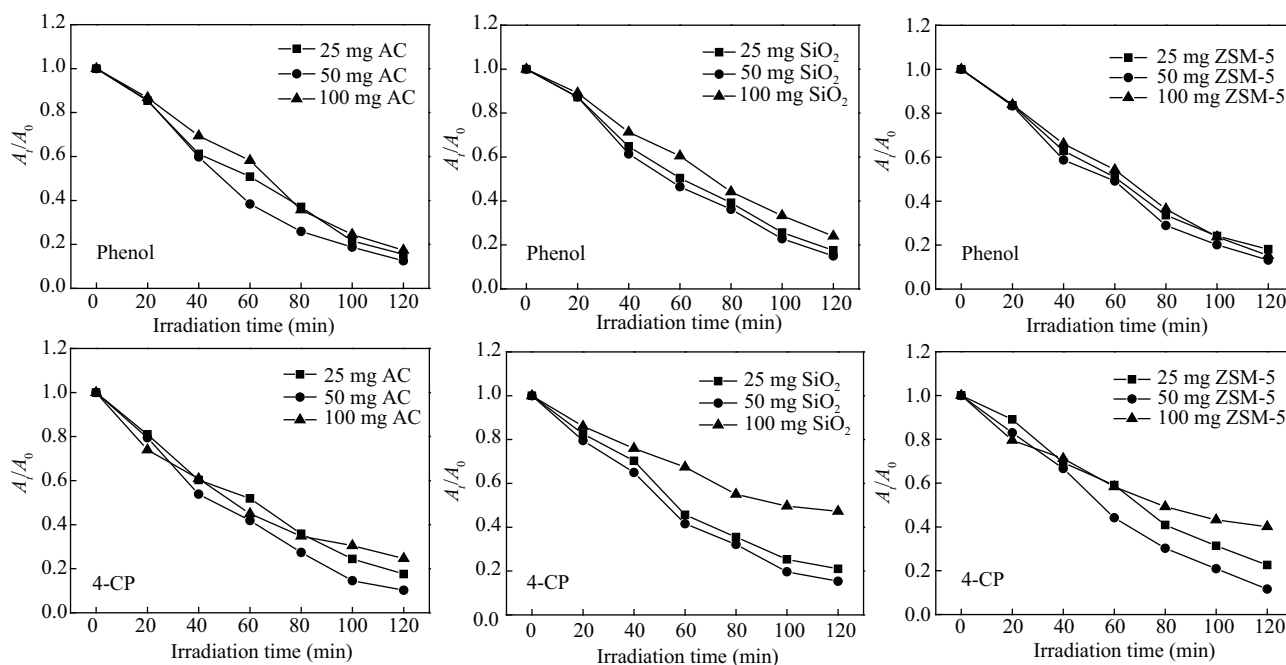
Electrons ( $e_{\text{cb}}^-$ ) in the conduction band on the TiO<sub>2</sub> surface can reduce molecular oxygen to superoxide anions. These superoxide anions are responsible for the generation of  $\bullet\text{OH}$  that has been indicated as the primary source of pollutant degradation (Galindo et al., 2000; Daneshvar et al., 2004; Konstantinou and Albanis, 2004).



The whole mechanism of photoactivity of TiO<sub>2</sub> particle is summarized in **Fig. 2**.

### 2.2 Effect of support concentration

To examine the effect of support on the degradation of phenol and 4-CP, a series of experimentation was accomplished by the different types of supports, namely AC, SiO<sub>2</sub> and ZSM-5. **Figure 3** illustrate the results for the photocatalytic degradation of phenol and 4-CP from aqueous solution utilizing different supports loading varying from 25 to 100 mg. It can be seen that photocatalytic degradation of phenol and 4-CP was found to increase then decrease with the increase in support concentration. This trend is probable for the reason that as the number of support particles surrounding the phenol and 4-CP increases, more phenol and 4-CP is adsorbed by these particles (Al-Asheh et al., 2003). It was found that for all three supports, an



**Fig. 3** Effect of  $\text{TiO}_2/\text{AC}$ , and  $\text{TiO}_2/\text{SiO}_2$ , and  $\text{TiO}_2/\text{ZSM-5}$  on absorption intensity of phenol and 4-CP.

optimal concentration of 50 mg with  $\text{TiO}_2$  produced the best photodegradation of phenol and 4-CP.

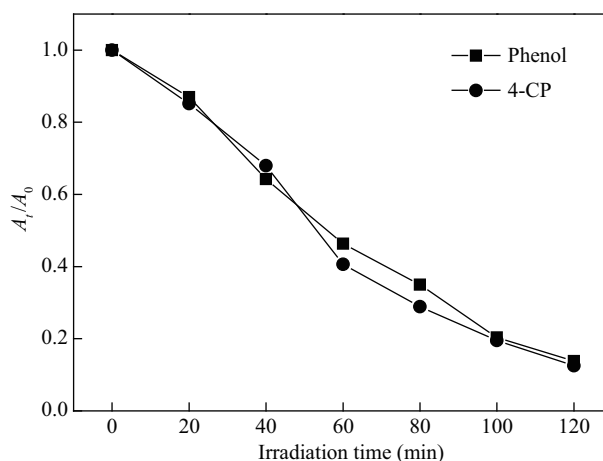
From **Fig. 3**, it can be concluded that all the supports when intermingle with  $\text{TiO}_2$  enhanced the photodegradation of phenol and 4-CP, with the order  $\text{AC} > \text{ZSM-5} > \text{SiO}_2$ .  $\text{TiO}_2$  aided with AC has the highest degradation efficiency for phenol and 4-CP under the experimental conditions. These results revealed that the effective surface area and adsorption capacity of the supported  $\text{TiO}_2$  were much higher than that of  $\text{TiO}_2$  alone, which favor rapid degradation of phenol and 4-CP (Bhattacharyya et al., 2004).

### 2.3 Comparison with rice husk

**Figure 4** shows the effect of RH on the absorption intensity of phenol and 4-CP versus irradiation time. It is easily seen from **Fig. 4** that relative to phenol and 4-CP degradation over  $\text{TiO}_2$  alone (**Fig. 1**), the degradation of phenol and 4-CP is fairly increased in the presence of RH. **Figure 5** shows that the synergistic result of  $\text{TiO}_2$  and RH is in comparison with  $\text{TiO}_2/\text{AC}$  and  $\text{TiO}_2/\text{ZSM-5}$  systems. This advocates that RH is a substitute and inexpensive substance and can be used as support or co-sorbent.

### 2.4 Kinetics of photodegradation of 4-NP

From engineering point of view, it is useful to find out a simple and user-friendly rate equation that fits the experimental rate data. It can be seen from **Fig. 3** that the dependence of phenol and 4-CP concentration on the irradiation time was fitted to an exponential function suggesting that the photodegradation of phenol and 4-CP documented pseudo first-order kinetics with respect to the



**Fig. 4** Change in absorption intensity of phenol and 4-CP with  $\text{TiO}_2/\text{RH}$ . RH: rice husk.

phenol and 4-CP concentration:

$$-\frac{d[\text{pollutant}]}{dt} = k_{\text{app}}[\text{pollutant}] \quad (9)$$

The integration of Eq. (9) under the restriction  $[\text{pollutant}]_t = [\text{pollutant}]_0$  at the start of irradiation ( $t = 0$ ), where  $[\text{pollutant}]_0$  is the initial concentration, yields Eq. (10):

$$-\ln\left(\frac{[\text{pollutant}]_t}{[\text{pollutant}]_0}\right) = k_{\text{app}} t \quad (10)$$

where,  $k_{\text{app}}$  ( $\text{min}^{-1}$ ) is the apparent first order rate constant. The computed  $k_{\text{app}}$  values from Eq. (10) at optimal support concentration (50 mg) are listed in **Table 1**. It can be seen that  $k_{\text{app}}$  values for  $\text{TiO}_2/\text{ZSM-5}$  and  $\text{TiO}_2/\text{RH}$  are

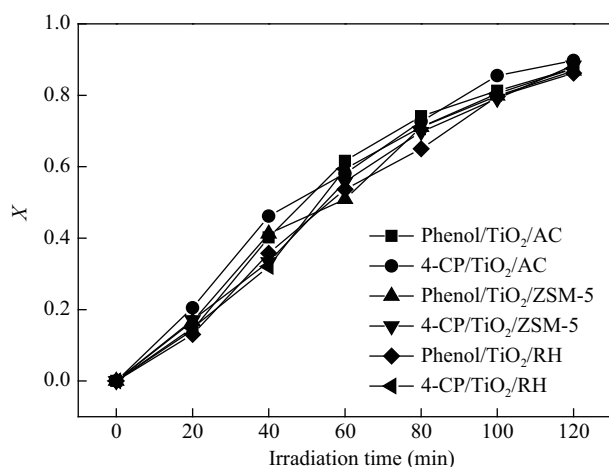


Fig. 5 Degree of degradation ( $X$ ) of phenol and 4-CP assisted by  $\text{TiO}_2$ .

in good agreement and slightly less than  $\text{TiO}_2/\text{AC}$  system, demonstrating that RH can be used as alternative supporting material to AC and ZSM-5.

Table 1 Apparent rate constant ( $k_{\text{app}}$ ,  $\text{min}^{-1}$ ) of phenol and 4-CP under different conditions

Phenolic compound	$\text{TiO}_2$	$\text{TiO}_2/\text{AC}$	$\text{TiO}_2/\text{ZSM-5}$	$\text{TiO}_2/\text{SiO}_2$	$\text{TiO}_2/\text{RH}$
Phenol	0.0122	0.0181	0.0172	0.0159	0.0169
4-CP	0.0139	0.0195	0.0179	0.0163	0.0179

### 3 Conclusions

The photocatalytic degradation of phenol and 4-CP was investigated in aqueous suspensions of  $\text{TiO}_2$  irradiated by UV light. Different supporting materials were shown to enhance the photocatalytic degradation of phenol and 4-CP. All supports with  $\text{TiO}_2$  showed augmentation in degradation of phenol and 4-CP than that of bare  $\text{TiO}_2$  within 120 min of photocatalysis. The support 50 mg was found to be the most favorable concentration for degradation of phenol and 4-CP. In the degradation of phenol and 4-CP,  $\text{TiO}_2/\text{AC}$  system showed the highest photocatalytic efficiency as compared to that of other systems. A comparable result was obtained when an optimal amount of rice husk was used as a supporting material. Therefore, rice husk could be used as an alternative to AC and ZSM-5. This study will help noticeably an easy approach in enhancing the degradation of organic pollutants.

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