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Photocatalytic removal of Cr(VI) with illuminated TiO_2

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ABSTRACT

The effect of dissolved oxygen on the photocatalytic reduction of Cr(VI) with illuminated TiO_2 was studied in this work with variation of the solution pH, contact time, and initial Cr (VI) concentration. Oxygen or nitrogen gas was used as a purging gas. Overall, the removal efficiency of Cr(VI) decreased as the solution pH increased. The removal of Cr(VI) by UV/TiO_2 increased by decreasing the solution pH because of the increased potential difference between the conduction band of TiO_2 and Cr(VI)/Cr(III) as well as the anionic-type adsorption of Cr(VI) onto the TiO_2 surface. The removal efficiency of Cr(VI) increased in purging of nitrogen gas compared to that of oxygen gas because of less competition between dissolved oxygen and Cr(VI) in the reaction with the electron in the conduction band of TiO_2. The reduction pattern was better described by the first-order kinetic model.

Keywords: Dissolved oxygen; Photocatalysis; Cr(VI); TiO_2

1. Introduction

Chromium is widely used for several industrial purposes such as in metal processing, industrial plating, textiles, and leather [1,2]. The fate of chromium within the environment is closely related to its chemistry. Chromium found in industrial wastewater is both Cr(VI) and Cr(III). Compared with the Cr(III), Cr (VI) is very toxic, carcinogenic and mutagenic for human and environment apart from being mobile to a large extent [3–8]. Therefore, Cr(VI) reduction to Cr (III) is highly required in order to reduce the toxicity of chromium as well as to retard its mobility. The general processes for the treatment of industrial wastewater containing organics and metallic elements are ion exchange, reverse osmosis, direct precipitation, and adsorption with activated carbon. These methods require a greater exposed liquid surface area and long detention periods. Besides these, most of these methods need high capital cost and recurring expenses such as chemicals. These factors act as a huge burden on the management of a small-scale industrial facility [9–12].

To conquer these problems, the development of new or cheap adsorbents instead of the above methods as well as the application of advanced oxidation processes (AOP) have attracted much attention as talented techniques [13–28]. Overall AOPs, generating hydroxyl radical (·OH), have been widely used to destroy organic compounds that cannot be oxidized by conventional oxidants such as oxygen, ozone, and chlorine [17,20–23]. The hydroxyl radical can be

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generated in aqueous solutions using O_3/UV, H_2O_2/UV, Fe(II)/H_2O_2, TiO_2/UV, and ZnO/UV [20,21, 24–28]. Among these methods, the photocatalytic reaction using TiO_2/UV can simultaneously treat organic compounds and heavy metals that are susceptible to the redox reaction. Consequently, this method can be used as a pre- or posttreatment process in wastewater treatment because of its easiness of installation in conventional wastewater treatment facilities. The widely used form of TiO_2 was powder or thin film in the water treatment process [29–32]. During the photocatalytic reaction with TiO_2, separation efficiency of electron in conduction band and positive hole in valence band affect the photocatalytic efficiency of the contaminants. Photocatalytic reduction of Cr(VI) has been widely investigated in the presence of dissolved oxygen by Lin et al. [33].

This study investigated the effect of dissolved oxygen on the photocatalytic reduction of Cr(VI) with variation of the solution pH, contact time, and initial Cr(VI) concentration. In addition, kinetic parameters were obtained by application of zero-, first-, and second-order equations.

2. Methodology

2.1. Materials

P-25TiO_2 (a 70/30 mixture of anatase and rutile) was obtained from Degussa Corp. It has approximately a spherical and nonporous shape with greater than 99.5% purity. The specific surface area of the TiO_2 particles was $50 \pm 15\,m^2/g$ according to Evonik Industries. The average particle size of the TiO_2 particles was 21 nm. All chemicals were of analytical grade. $K_2Cr_2O_7$ purchased from Merck & Co was used to prepare the stock solution (1,000 mg/L) of Cr(VI). Experimental solutions of the desired concentrations were prepared by successive dilutions. All solutions were prepared with deionized water (18 M-cm) using a Hydro-Service reverse osmosis/ion-exchange apparatus. All bottles and glassware were acid-washed and rinsed with deionized water before use.

2.2. Experimental setup

The experimental setup for the photocatalytic reactor used in this work is schematically shown in Fig. 1. A 125 W medium-pressure UV lamp emitting maximum wavelength at 247.3 nm and light intensity equal to 1,020 μw/cm^2 (according to Shokofan Tosee Company in Iran) used as the radiation source. The reactor consists of two compartments: the outer one contained a 2 L solution and the inner one housed a UV lamp. The inner compartment was water-jacketed to maintain the solution temperature at 25℃ for all experiments. All photocatalytic experiments were performed with 1,000 mL solution. During experiments, the test solution in the reactor was constantly stirred. Oxygen or nitrogen gas was used at a purging gas rate of 2 L/min.

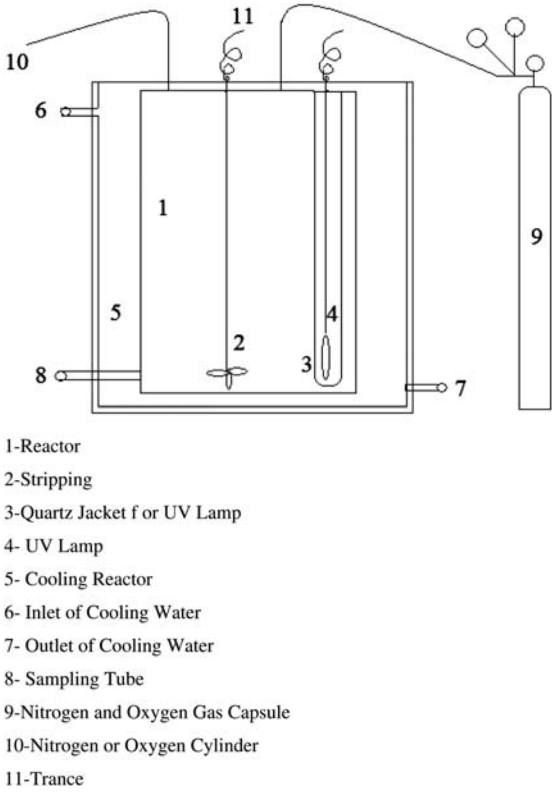

Fig. 1. A schematic diagram of the experimental setup for photocatalysis.

2.3. Photocatalytic experiments

Photocatalytic experiments were performed with variation of the solution pH (4, 6, 8), contact time (15–120 min), and initial Cr(VI) concentration (10, 20, 30, 40, 50 mg/L) in the purging of oxygen and nitrogen gas. TiO_2 dosage equal to 1 g/L was used for all experiments. The initial solution pH was adjusted by adding 0.1 M NaOH and HCl. All experiments were performed under ambient conditions for 2 h. TiO_2 suspensions were equilibrated in the dark for 30 min. After the equilibration period, the UV lamp was turned on and a portion of suspension was periodically taken from the reactor. All the samples were covered by aluminum foil to avoid ambient light. All experiments were accomplished at constant temperature (25 ± 2℃).

The sample suspensions were centrifuged at 4,000 rpm for 30 min to remove TiO_2 nanoparticles and then the residual Cr(VI) concentration in the supernatant was analyzed. Cr(VI) was analyzed by the 1,5-diphenylcarbazide method using a spectrophotometer (Shimadzu UV-160A) at a wavelength of 540 nm [34]. The removal efficiency of Cr(VI) was calculated by Eq. (1):

$$\mathrm{RE}\ (\%) = \left(\frac{C_0 - C}{C_0}\right) \times 100 \tag{1}$$

where C_0 and C are the initial and residual Cr(VI) concentrations at time t in solution, respectively.

3. Results and discussion

3.1. Removal of Cr(VI) in purging of different gases

The effect of the concentration of dissolved oxygen on the removal of 10 mg/L Cr(VI) with illuminated TiO_2 was investigated at constant catalyst dosage (1 g/L) and by varying the initial solution pH (4, 6, or 8) at different time intervals. Fig. 2a and 2b shows the removal of Cr(VI) by TiO_2/UV in the continuous purging of oxygen and nitrogen gas (each 2 L/min), respectively, at different solution pH. At initial condition (before turning on the UV light), the removed fraction of Cr(VI) ranged from 0.1 to 18.0% in purging of oxygen gas, while it ranged from 29 to 73% in purging of nitrogen gas. The different removal of Cr (VI) with variation of pH observed at initial condition in purging of either O_2 or N_2 gas can be explained by an anionic-type adsorption behavior of Cr(VI) onto TiO_2. The pH at zero point of charge (pH_{zpc}) of P-25 TiO_2 is reported to be 6.5 [35]. Therefore, it is reasonable to explain that anionic species of Cr(VI), such as CrO_4^{2-} and $HCrO_4^{-}$, can be easily adsorbed onto the TiO_2 surface below the pH_{zpc}. However, a greater removal percentage of Cr(VI) observed in purging of N_2 gas than in purging of O_2 gas is difficult to explain.

As the reaction progressed, a greater photocatalytic removal of Cr(VI) was observed at lower solution pH, due to an increased potential difference between the conduction band of TiO_2 and Cr(VI)/Cr(III) as well as the anionic-type adsorption of Cr(VI) onto the surface of TiO_2. As discussed previously by the other researcher, thermodynamic driving force for the reduction of Cr(VI) is related to the potential difference between the conduction band of TiO_2 and the heavy metal ions [36]. The removal of Cr(VI) increased as the reaction time progressed in purging of either O_2 or N_2 gas. Fig. 3 shows a profile of the concentration of dissolved oxygen in purging of O_2 with variation of time. The concentration of dissolved oxygen decreased from 8.67 to 2.93 mg/L from a reaction time of 15 to 120 min. The concentration of dissolved oxygen in the TiO_2 suspension in purging of N_2 gas was below 0.1 ppm over all the reaction time (data not shown).

A complete removal of Cr(VI) was observed after 105 min at pH 4 in purging of nitrogen gas. Competition between Cr(VI) and oxygen to react with an electron in conduction band of TiO_2 may explain this trend [25].

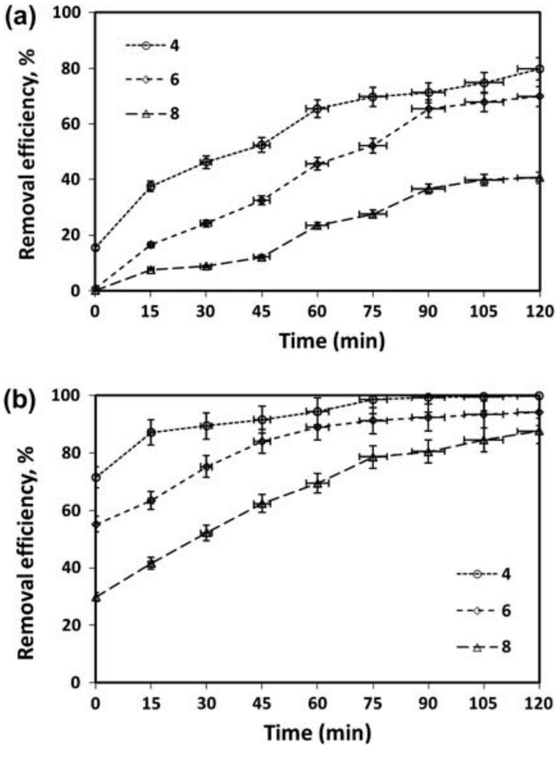

Fig. 2. Effect of pH on the removal of Cr(VI) in purging of (a) oxygen and (b) nitrogen gas (TiO_2 = 1 g/L, Cr(VI) = 10 mg/L).

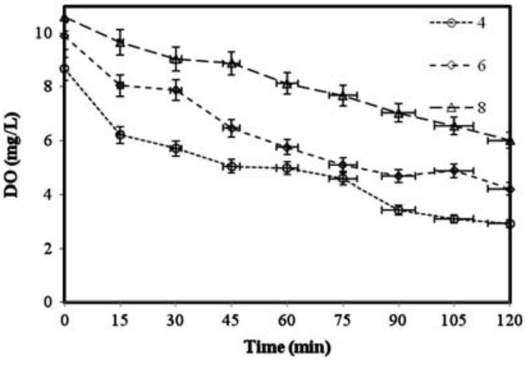

Fig. 3. Variation of the concentration of dissolved oxygen at different pH during the photocatalytic reaction in purging of oxygen.

$$O_2 + 2H_2O + 4e^- \xrightarrow[hv]{TiO_2} 4OH^- \quad (2)$$

$$CrO_4^{2-} + 4H_2O + 3e^- \rightarrow Cr(OH)_3 + 5OH^- \quad (3)$$

The experimental results obtained in purging with two different gases and at different solution pHs indicate that the reduction rate of Cr(VI) greatly depends on both the solution pH and the concentration of dissolved oxygen. However, negligible effect of the concentration of dissolved oxygen on the reduction of Au (III) and Cr(VI) was reported by Borgarello [37] and Lin [33], respectively, during the UV/TiO_2 photocatalytic reaction.

It can be assumed that the competition between dissolved oxygen and metal ion to capture electron in the conduction band of TiO_2 may depend on the type of metal ion. Thus, metal ions such as Cr(VI) (E^o = +1.33 V) and Au(III) (E^o = +1.498 V) having a higher redox potential can easily be reduced, especially at acidic conditions. While the reduction of metal ions having a lower redox potential such as Hg(II) and Cu (II) is difficult in the presence of dissolved oxygen due to greater competition between these metal ions and electron in the conduction band of TiO_2.

3.2. *Removal of Cr(VI) at different concentration of Cr(VI)*

The removal of Cr(VI) by TiO_2/UV was investigated with the variation of initial Cr(VI) concentration (10, 20, 30, 40, 50 mg/L) at initial pH 4 and at constant TiO_2 dosage (1 g/L). Fig. 4 shows that, for all concentrations, a rapid increase in the removal of Cr(VI) was observed up to 30 min and then the slope was gradually reduced. Percent removal of Cr(VI) decreased as the initial Cr(VI) concentration increased. The presumed reason is that when the initial Cr(VI) concentration increased, more Cr(VI) molecules can be removed on the surface of TiO_2. The large amount of removed chromium might have an inhibitive effect on the further photocatalytic reaction of Cr(VI) because of the decreased adsorption sites on the TiO_2 as well as the limited oxidants on the surface of TiO_2 [7,38,39].

3.3. *Kinetics*

In order to obtain the kinetic information, the experimental results in Fig. 4 were fitted with zero-, first-, and second-order equations. From this evaluation, the reduction pattern was better described by the first-order kinetic model. Because the initial concentration of Cr(VI) employed in this study was low, the

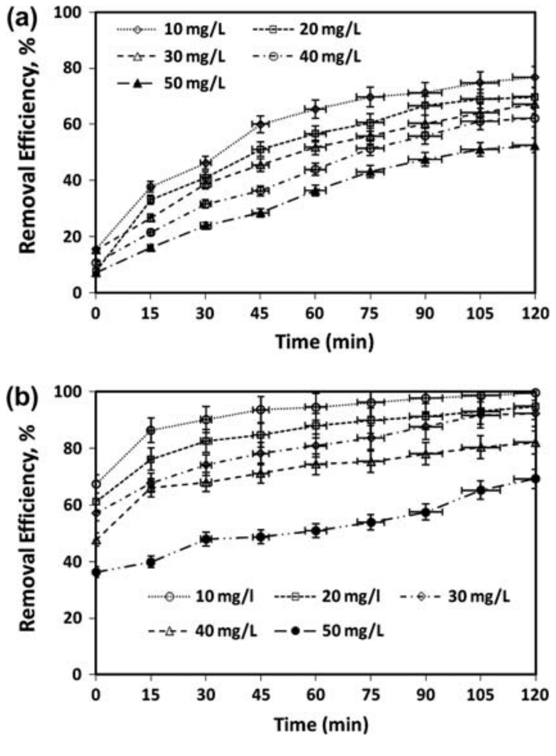

Fig. 4. Effect of initial Cr(VI) concentration on the removal of Cr(VI) in purging of (a) oxygen and (b) nitrogen gas (pH = 4, TiO_2 = 1 g/L).

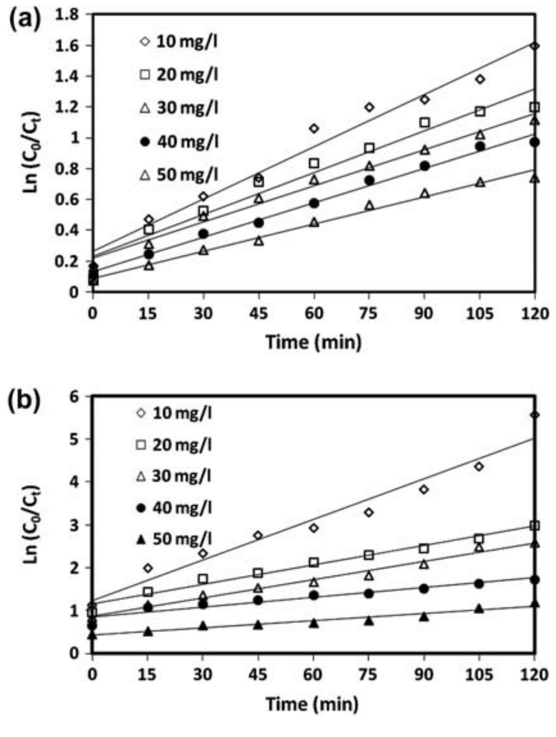

Fig. 5. First-order kinetic model for Cr(VI) removal in purging of (a) oxygen and (b) nitrogen gas (pH = 4, TiO_2 = 1 g/L).

Table 1
Kinetic parameters for the photocatalytic removal of Cr(VI) in purging of oxygen gas at different initial Cr(VI) concentrations ($TiO_2 = 1$ g/L, pH = 4)

Cr(VI) (mg/L)	Zero-order		First-order		Second-order	
	k_0		k_1		k_2	
	(mol L^{-1} min^{-1})	R^2	(min^{-1})	R^2	(L mol^{-1} min^{-1})	R^2
10	0.0485	0.9031	0.0113	0.9759	0.0026	0.9922
20	0.1726	0.9676	0.009	0.9552	0.001	0.9901
30	0.1224	0.9411	0.0078	0.9877	0.0005	0.998
40	0.0925	0.8673	0.0074	0.9918	0.0003	0.988
50	0.1936	0.9714	0.0059	0.9888	0.0002	0.9914

Table 2
Kinetic parameters for the photocatalytic removal of Cr(VI) in purging of nitrogen gas at different initial Cr(VI) concentrations ($TiO_2 = 1$ g/L, pH = 4)

Cr(VI) (mg/L)	Zero-order		First-order		Second-order	
	k_0		k_1		k_2	
	(mol L^{-1} min^{-1})	R^2	(min^{-1})	R^2	(L mol^{-1} min^{-1})	R^2
10	0.0197	0.7796	0.0441	0.9571	0.4743	0.5789
20	0.0461	0.8144	0.0151	0.9741	0.0063	0.9371
30	0.0818	0.9386	0.0142	0.9835	0.003	0.8998
40	0.0291	0.9684	0.0076	0.9225	0.0007	0.9739
50	0.091	0.8097	0.0056	0.9435	0.0003	0.8956

adsorption kinetics can be described adequately by a simplified first-order rate equation (Eq. (4)) [40].

$$\ln[C_0/C_t] = k_1 t \tag{4}$$

where C_0 and C_t are the Cr(VI) concentrations at initial stage and at time t, respectively. k_1 is the rate constant and t is the reaction time. Fig. 5a and 5b shows the plot of $\ln[C_0/C_t]$ vs. t for the reduction of Cr(VI) by TiO_2/UV. The k values were obtained from the slope of the linear regression. First-order rate constants including zero- and second-order rate constants at different concentrations of Cr(VI) in the purging of oxygen and nitrogen gas are summarized in Table 1 and Table 2, respectively. As the concentrations of both Cr(VI) increased, the rate constant in both systems decreased.

4. Conclusions

The major findings of this study are as follows:

The removal of Cr(VI) by UV/TiO_2 increased by decreasing pH because of the increased potential difference between the conduction band of TiO_2 and Cr(VI)/Cr(III) as well as the anionic-type adsorption of Cr(VI) onto the TiO_2 surface. Another reason for the decreasing removal rate at higher pH may be the increased deposition of $Cr(OH)_3$, which was produced from Cr(VI) reduction on the catalyst surface. The removal efficiency of Cr(VI) decreased as the initial Cr(VI) concentration increased. The presumed reason is that when the initial Cr(VI) concentration increased, the more reduced Cr(III) occupy the limited surface of TiO_2. The reduction pattern was better described by the first-order kinetic model. Finally, dissolved oxygen shows a greater inhibition effect on the reduction of Cr(VI) than the nitrogen gas which shows a noticeable reduction.

Acknowledgments

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