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# Photocatalytic degradation of 2-propanol by using $Pt/TiO_2$ prepared by microemulsion technique

Sumaeth Chavadej [a,*], Pattamawadee Phuaphromyod [a], Erdogan Gulari [b],
Pramoch Rangsunvigit [a], Thammanoon Sreethawong [a]

[a] *The Petroleum and Petrochemical College, Chulalongkorn University, Soi Chula 12, Phyathai Road, Pathumwan, Bangkok 10330, Thailand*
[b] *Department of Chemical Engineering, The University of Michigan, Ann Arbor, MI 48109, USA*

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## Abstract

The photocatalytic degradation of 2-propanol was studied by using platinum, titania, and platinum loaded on titania as photocatalysts. In this study, the catalysts were prepared by microemulsion technique, which gives ultrafine particles in the range of 3–5 nm. Each synthesized photocatalyst was suspended in an aqueous solution of 2-propanol, and the solution was photoirradiated by an 11 W low pressure mercury lamp. The results showed a negligible activity of platinum and a relatively low activity of titania in the degradation of 2-propanol, but a significantly high activity of platinum loaded on titania was observed. The effects of the influencing factors, including initial concentration of 2-propanol, catalyst dosage, initial pH, and dissolved oxygen, were studied. The results revealed that the most efficient degradation of 2-propanol was achieved in a highly acidic region, and the degradation rate increased with both catalyst dosage and initial concentration of 2-propanol. The dissolved oxygen was found to have a significant influence affecting the photocatalytic degradation of 2-propanol since it acts as an electron scavenger and is responsible for producing hydroxyl radical. Acetone was detected as an intermediate during the photocatalytic reaction of 2-propanol.

*Keywords:* Photocatalysis; 2-Propanol; Titania; Platinum; Microemulsion

## 1. Introduction

Under light illumination conditions, various heterogeneous photocatalysts offer an attractive means of destroying most organic pollutants present in water at low concentrations. $TiO_2$-based photocatalysis has been widely studied as an alternative to the conventional processes such as ozonation and chlorination for elimination of toxic and hazardous organic substances in water and air [1–3]. This photocatalytic process has been demonstrated as a potential means for complete mineralization of organic wastes at a low cost and with simplicity in operation. Moreover, it does not utilize toxic materials, and it can be operated at ambient conditions. The fundamental aspects and potential advantages of photocatalytic remediation processes have been described in details [4,5]. The basic principle of photocatalysis is relatively simple; when a semiconductor photocatalyst is exposed to light with appropriate photon energy, the electrons in the valence band, which are excited, will migrate to the conduction band, leaving behind positive holes [6]. The positive holes react with water and/or oxygen to produce hydroxyl radicals, which are a primary oxidizing species [7,8]. In addition, the direct oxidation can occur by the adsorption of organic substrate at the positive holes on the catalyst surface. Increasing adsorption of the organic substrate onto the catalyst surface can enhance the photocatalytic reaction rate by increasing the probability of oxidation reaction to be greater than the expense of the electron–hole recombination reaction. Platinum, as a co-catalyst, has been incorporated with $TiO_2$ to increase the adsorption of organic pollutants [9,10]. In addition to the increase in the adsorption of organic molecules, it has been postulated that a small amount of platinum accelerates the formation of superoxide radicals, leading to decreasing the rate of electron–hole recombination [11,12]. However, a high concentration of platinum is detrimental to the oxidation reaction because the platinum particles instead act as recombination centers [1,13].

The use of ultrafine particles of various metals as catalysts has been found in a number of industrial applications.

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* Corresponding author. Tel.: +66 2 218 4139; fax: +66 2 218 4139.
*E-mail address:* sumaeth.c@chula.ac.th (S. Chavadej).

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As widely known, several techniques have been developed to synthesize various types of nanoparticles with any desired properties. Microemulsion technique is a novel method to prepare ultrafine particles because of the ability to control the size of particle formed and prevent the aggregation [14]. In this study, a water-in-oil (W/O) microemulsion known as Winsor Type II microemulsion was selected for catalyst preparation because this technique can provide nanosized particles, which are much smaller than an oil-in-water (O/W) or Winsor Type I microemulsion. The procedure to prepare the ultrafine particles by this W/O microemulsion technique starts with two identical W/O microemulsions. One system dissolves a reactant whereas the other one dissolves another reactant in the aqueous cores. After mixing these two microemulsion systems, both reactants come to react with each other due to collision and coalescence of the tiny liquid droplets (reverse micelles) suspended in the microemulsion, and the particles are produced in the aqueous cores. However, the way to achieve this preparation process can be also done by another well-known procedure. This is the method of adding a reducing agent or precipitating agent into the W/O microemulsion containing the reactant in the aqueous cores. It was first reported in the synthesis of Pd, Rh, and Pt by reducing the corresponding salts in the aqueous cores of W/O microemulsion with hydrazine or hydrogen gas [15]. This microemulsion technique can be employed to produce very fine particles of catalysts, which are efficient for photocatalytic processes [14,16].

In this study, platinum nanoparticles in the range of 3–5 nm were synthesized using the microemulsion technique. The aim of this work was to determine the effects of platinum loading, substrate concentration, catalyst concentration, solution pH, and dissolved oxygen on the photocatalytic oxidation of 2-propanol or *iso*-propanol, a representative organic pollutant, under the illumination of UV light.

## 2. Experimental

### 2.1. Photocatalyst preparation

Analytical grade heptane ($C_7H_{16}$) from Lab-Scan Asia was used as a continuous phase in W/O microemulsions. Bis(2-ethylhexylsulfosuccinate) or AOT ($C_{20}H_{37}NaO_7S$), an anionic surfactant, was supplied by Fluka Chemicals. Chloroplatinic acid ($H_2PtCl_6$) and commercial $TiO_2$ obtained from Fluka Chemicals were used as a precursor for platinum particles and a supporting material, respectively. Hydrazine hydrate ($N_2H_4{\cdot}H_2O$) from Carlo Erba Reagent, was used as a reducing agent. Analytical grade acetone from Lab-Scan Asia was used to break microemulsion in order to separate the synthesized nanoparticles.

The nanosized Pt particle preparation method used in this study basically followed that of Pillai and Shah [15]. The formation of a W/O microemulsion (Winsor Type II) was done by dissolving an appropriate amount of AOT into 50 ml of heptane. A quantity of 1 ml of aqueous solution containing 0.05 M of chloroplatinic acid was added dropwisely into the AOT/heptane phase. The mixture was mixed vigorously, and then an amount of 1 ml of 0.5 M hydrazine solution was added dropwisely into the mixture. The solution was well mixed and left around 24 h at room temperature (25–27 °C) to allow the reducing agent react with the precursor completely. The transformation of the precursor to platinum particles could be observed by the change in the solution color from slightly yellow to black. The platinum particles formed were then separated from the microemulsion system by adding a small amount of acetone to break the microemulsion. Consequently, the platinum particles then precipitated out from the solution. The precipitate was washed by heptane, acetone, and hot distilled water to remove all remaining chemicals. The platinum nanoparticles were obtained by centrifuging the washed precipitate at 4000 rpm for 10 min, dried at 383 K for 12 h, and finally calcined at 573 K for 3 h to remove the adsorbing surfactant after the separation step. To prepare 0.23% Pt-loaded $TiO_2$, 4.2 g of $TiO_2$ was added to the microemulsion before the step of the hydrazine addition in order to allow the platinum nanoparticles deposit on its surface. After the use of acetone to break the microemulsion, the precipitate of Pt-loaded $TiO_2$ was washed, separated, dried, and calcined in the same manner as above explained. For the sake of comparisons, blank $TiO_2$ was also heat-treated at 573 K for 3 h before use as photocatalyst in reaction experiments.

### 2.2. Photocatalyst characterizations

The surface areas of all prepared catalysts were determined by a BET surface area analyzer (Quantachrom, Autosorb-1) using nitrogen adsorption analysis. A catalyst sample was dried and outgassed in a sample cell at 200 °C for at least 4 h before adsorption. The specific area of each catalyst was calculated from the 5-point adsorption isotherm. The results were analyzed by using Autosorb ANAGAS software, Version 2.10. The crystalline phases of the catalysts were investigated by an X-ray diffractometer (Rigaku, RINT-2200) equipped with a graphite monochromator and a Cu tube for generating CuKα radiation ($\lambda = 1.5406$ Å) at a generator voltage of 40 kV and a generator current of 30 mA. A nickel filter was used as the Kα filter. The goniometer parameters were; divergence slit = $1^\circ(2\theta)$, scattering slit = $1^\circ(2\theta)$, and receiving slit = 0.3 mm. The catalyst sample was held on a glass slide holder and was examined in the $2\theta$ range of 5–80° at a scanning speed of $5^\circ(2\theta)\,\mathrm{min}^{-1}$ and a scan step of $0.02^\circ(2\theta)$. The digital output of the proportional X-ray diffractometer and the goniometer angle measurements were sent to an online microcomputer to record the data and to perform subsequent analyses. The particle sizes of all prepared catalysts were measured by using a transmission electron microscope (TEM) (Philips, 200 CX).

### 2.3. Photocatalytic experiments

Analytical grade 2-propanol or *iso*-propanol ($C_3H_8O$), supplied by Carlo Erba Reagent was used as a reaction substrate in this study. Solution pH was varied by using nitric acid ($HNO_3$) and sodium hydroxide (NaOH). Ultra-high pure nitrogen, air, and oxygen gases were used to vary the dissolved oxygen concentration during the photocatalytic experiment. A dissolved oxygen (DO) meter (Orion model 860) was used to measure the value of the dissolved oxygen in the solution.

A photocatalytic unit used in this experiment was operated as a batch reactor. The apparatus consisted of a cylindrical glass vessel containing 80 ml of the substrate solution. The reactor was wrapped with aluminum foil to prevent the loss of UV light. The top of the reactor was closed to avoid any vaporization. The reactor was immersed into a water bath for maintaining a reaction temperature at 25 °C. A magnetic stirrer was used to induce continuous mixing of the reaction solution throughout the experimental period. An 11 W low pressure mercury lamp (Philips) with wavelength in the range of 200–300 nm was dipped into the reaction solution in order to illuminate the solution thoroughly. The experiment was carried out at different 2-propanol concentrations, catalyst loadings, initial solution pH values, and dissolved oxygen levels. The reaction was then started when the UV lamp was turned on. Solution samples were taken for every 90 min interval until 360 min for both qualitative and quantitative analyses by means of a gas chromatograph (Perkin-Elmer, Carbowax 20 M column, $N_2$ carrier gas) equipped with a head-space auto sampling and flame ionization detector (FID).

## 3. Results and discussion

### 3.1. Photocatalyst characterization results

The BET specific surface areas of the $TiO_2$ and 0.23% Pt/$TiO_2$ are 10.48 and 8.89 $m^2/g$, respectively. The crystal structures of the studied catalysts identified by XRD patterns are shown comparatively in Fig. 1. Both catalysts show the dominant anatase peaks at $2\theta = 25°$, 38°, 48°, 54°, and 55° and a small fraction of rutile at $2\theta = 27°$. It is worth noting that no peak of platinum at $2\theta = 40°$ was observed in the case of Pt/$TiO_2$, suggesting that the microemulsion technique yields highly dispersed platinum particles on the $TiO_2$ photocatalyst. The particle sizes of the prepared catalysts were measured from the TEM micrographs. The TEM micrograph of the Pt particles is illustrated in Fig. 2. The particle size of the Pt particles was measured to be around 3–5 nm. Fig. 3 shows the TEM micrograph of Pt catalyst loaded commercial $TiO_2$. The picture shows the good dispersion of tiny platinum particles onto the surface of the commercial $TiO_2$ particles. As shown in Fig. 3, the larger boundary represents commercial $TiO_2$ with the size distribution of around 100–150 nm in diameter, and the uniformly distributed dark patches on the surface of the $TiO_2$ particles are platinum. These two types of the particles, i.e., $TiO_2$ and Pt particles, can be clearly distinguished because of their different electron density during being exposed with electron beam.

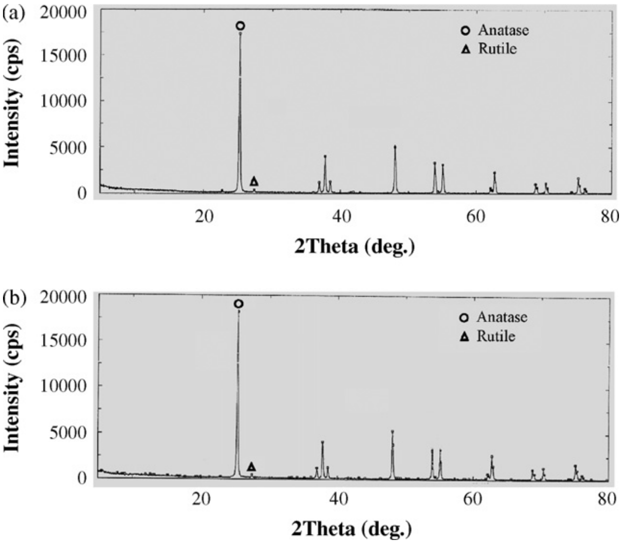

Fig. 1. XRD patterns of (a) commercial $TiO_2$ and (b) platinum-loaded $TiO_2$.

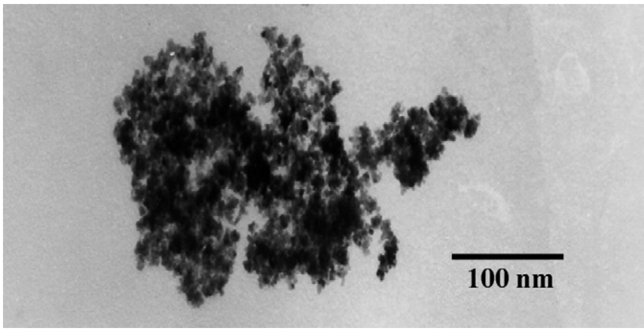

Fig. 2. TEM micrograph showing a large cluster of tiny platinum particles.

### 3.2. Effect of the presence of photocatalysts on the 2-propanol degradation

Fig. 4 illustrates the profiles of 2-propanol degradation by using different catalysts (Pt, $TiO_2$, and Pt/$TiO_2$) compared with the degradation without catalyst (photolysis). The results showed that the presence of Pt alone did not significantly enhance the photocatalytic activity as compared to the system without catalyst (photolysis). In contrast, the presence of commercial $TiO_2$ enhanced the degradation of 2-propanol. The explanation is that in the case of Pt alone, Pt has a continuum between valence band and conduction band (no band gap) due to its non-semiconductor property resulting in impossibility for the $e^-/h^+$ separation during light illumination, so the $e^-/h^+$ pair produced by photoirradiation can recombine easily, reducing the chance to react with other species [4]. In contrast, under UV illumination on $TiO_2$, its semiconductor property causes the excited

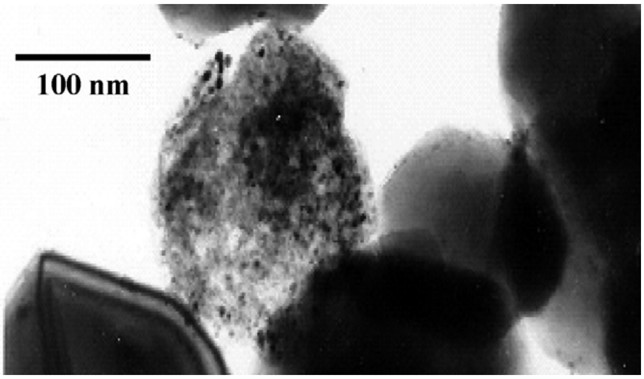

Fig. 3. TEM micrograph of platinum nanoparticles loaded on commercial $TiO_2$.

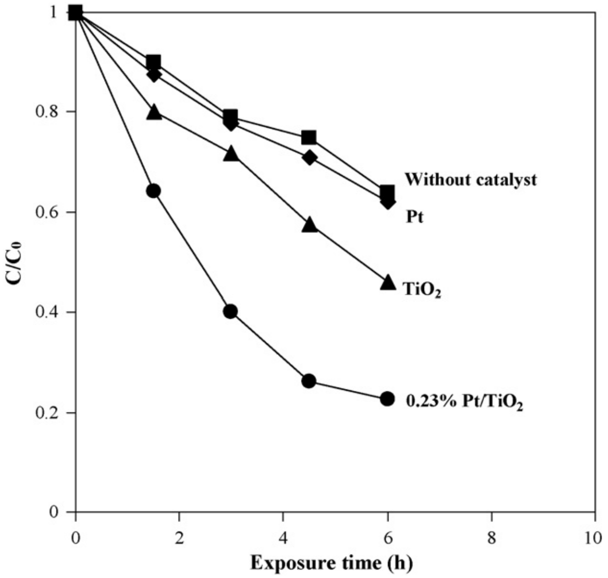

Fig. 4. Photocatalytic degradation of 2-propanol by using different types of catalysts under UV illumination (catalyst dosage, 0.2 g/l; initial 2-propanol concentration, 1.3 mM; initial solution pH, 7; initial dissolved oxygen, ∼7 mg/l; temperature, 45 °C).

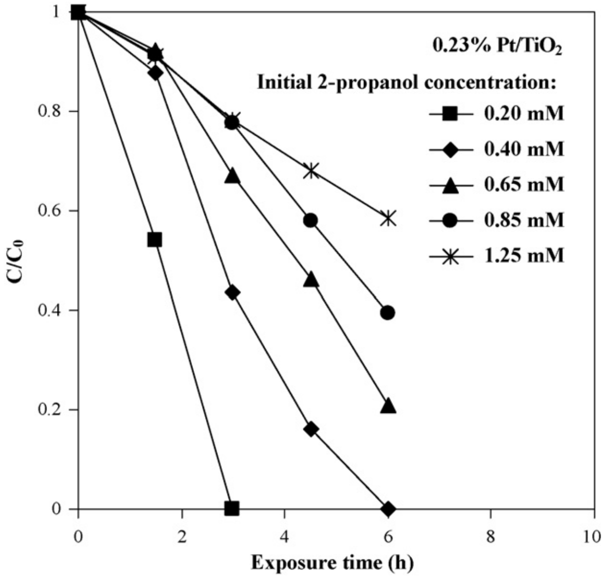

Fig. 5. 2-Propanol concentration ratio as a function of exposure time under different initial 2-propanol concentrations (catalyst dosage, 0.5 g/l; initial solution pH, 7; initial dissolved oxygen, ∼7 mg/l; temperature, 25 °C).

electron moving across the band gap from the valence band to the conduction band, leading to the separation of the $e^-/h^+$ pair and further the hydroxyl radical formation induced by these photogenerated species. Subsequently, the hydroxyl radical produced will then be able to react with 2-propanol. Interestingly, the degradation was substantially enhanced in the presence of $Pt/TiO_2$ as compared to the blank since the Pt catalyst provides the adsorption sites for 2-propanol, leading to increasing the degradation of 2-propanol [17]. In addition, Pt is believed to act as electron sink for better separation of the $e^-/h^+$ pairs generated by the band gap excitation of $TiO_2$ under the UV illumination and also help reduce the activation energy of photocatalytic reaction [18].

### *3.3. Effect of influencing parameters on the photocatalytic degradation*

Several parameters affecting the photocatalytic degradation of organic compounds present in water are light intensity, initial concentration of reaction substrate, type of catalyst, amount of catalyst, temperature, solution pH, dissolved oxygen, and electrolytes [19,20]. In this study, the effects of four parameters, including initial 2-propanol concentration, catalyst dosage, initial pH, and dissolved oxygen, were determined. Since $Pt/TiO_2$ was the most effective catalyst in degrading 2-propanol, this catalyst was then employed in further experiments.

#### *3.3.1. Effect of initial concentration of 2-propanol*

Fig. 5 shows the effect of the initial concentration of 2-propanol on the profiles of 2-propanol degradation in the presence of $Pt/TiO_2$. The effect of initial 2-propanol concentration on the degradation rate can be explained for two limiting conditions. First, at a given catalyst dosage and light intensity, the $e^-/h^+$ pair and reactive species are adequately formed at a constant rate, if the initial 2-propanol concentration is low. At a low initial 2-propanol concentration, the degradation rate (using the method of half-lives by the initial slope obtained from the plot of concentration and half-time, $t_{1/2}$, required for the concentration to decrease to one-half of its initial value) increases with increasing 2-propanol concentration. For the studied range of 2-propanol concentrations, the results follow this model as the degradation rate increases with the concentration. The other extreme is the case of insufficient hydroxyl radicals, which appears at very high 2-propanol concentrations. The degradation rate exhibits a maximum value, indicating that it is limited by the amount of reactive species produced by the UV illumination. At the highest 2-propanol concentration (1.25 mM) as shown in Fig. 5, the rate trends to be constant because of three involving factors. Firstly, the limitation of light illumination yields a certain amount of active hydroxyl radical to react with 2-propanol. Secondly, it may be due to the limit of dissolved oxygen in the system, which will be experimentally verified later. Finally, the higher the 2-propanol concentration, the higher concentrations of intermediate products is produced, which compete for reacting with hydroxyl radical. The formation of intermediate products was also investigated in this study and will be discussed later.

Under the studied conditions, the degradation rate shows a linear relationship fit to an empirical kinetics equation of zero-order, as shown in Fig. 5. It can be concluded that the rate of 2-propanol degradation does not depend on the initial concentration of 2-propanol under the studied conditions. It means that the rate of degradation may be limited by an active species, and the reaction at the surface is not fast enough so the initial 2-propanol concentration is not a significant factor affecting the degradation rate of 2-propanol.

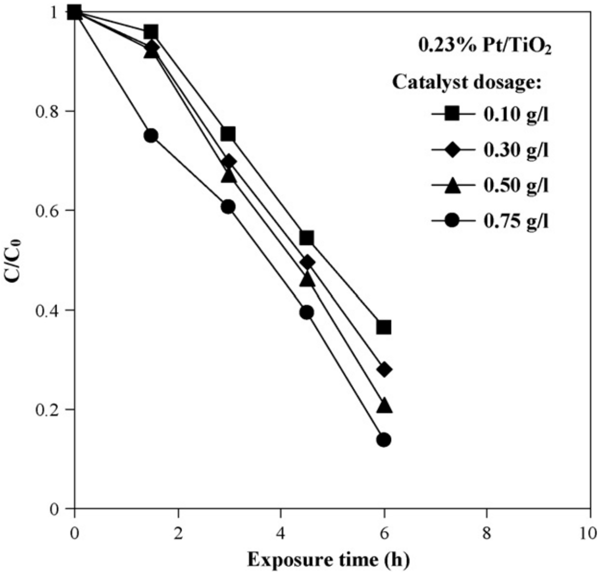

Fig. 6. 2-Propanol concentration ratio as a function of exposure time under different dosages of Pt/$TiO_2$ (initial 2-propanol concentration, 0.65 mM; initial solution pH, 7; initial dissolved oxygen, ~7 mg/l; temperature, 25 °C).

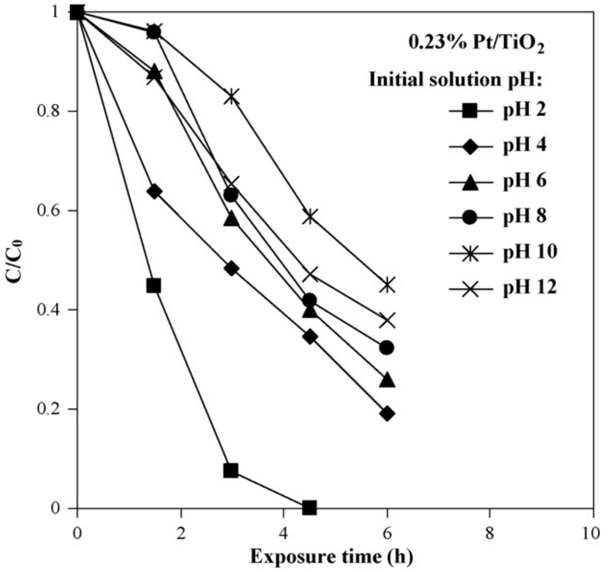

Fig. 7. 2-Propanol concentration ratio as a function of exposure time under different pH values (catalyst dosage, 0.5 g/l; initial 2-propanol concentration, 0.65 mM; initial dissolved oxygen, ~7 mg/l; temperature, 25 °C).

### *3.3.2. Effect of catalyst dosage*

To determine the effect of catalyst dosage, the 2-propanol concentration was fixed at 0.65 mM (39.25 mg/l) and the catalyst dosage was varied from 0.1 to 0.75 g/l. Fig. 6 shows the effect of catalyst dosage of Pt/$TiO_2$ on the 2-propanol degradation. The results showed that the reaction rate of 2-propanol degradation depended on the dosage of Pt/$TiO_2$. Increasing the catalyst dosage results in increasing the generation of active species to react with organic molecules, consequently increasing the reaction rate. The cooperation of light and catalyst is very important for the photocatalytic process. At a low initial 2-propanol concentration with a relatively high amount of catalyst, the dependence of the degradation rate on catalyst amount was not observed. In contrast, for a high concentration of 2-propanol, a use of an insufficient amount of catalyst leads to a low reaction rate, but an increase in catalyst dosage can increase the overall reaction rate. However, a large increase in either substrate concentration or catalyst dosage can cause the limitation of the light penetration that affects the photocatalytic reaction [8,21]. However, this study did not make an attempt to determine an optimum dosage of the catalyst.

### *3.3.3. Effect of initial solution pH*

Fig. 7 shows that the 2-propanol degradation becomes much more efficient under acidic condition and decreases drastically with increasing pH, but the degradation rate increases again at pH around 12. It is interesting to note that the solution pH did not alter significantly during the studied degradation period. The results showed the dramatic effect of initial solution pH on the degradation of 2-propanol since pH simply influences the surface charge of the catalyst, affecting the adsorption of the substrate onto the surface of the catalysts known as reaction site as well as the hydroxyl radical ($OH^{\bullet}$) formation. As $TiO_2$ possesses an amphoteric surface, 2-propanol can adsorb preferentially on the positively charged $TiO_2$ surface in acidic condition. Thus, the direct oxidation of 2-propanol is predominant at a low pH. Under a very high pH condition (pH 12), the production rate of the reactive hydroxyl radical increases, which becomes more available for the 2-propanol degradation [22]. The explanation of the effect of a very high pH will be discussed again later.

### *3.3.4. Effect of dissolved oxygen*

In order to determine the effect of dissolved oxygen on the 2-propanol photocatalytic degradation, the experiment was conducted at different dissolved oxygen levels. First, the experiment was carried out in the deaerated condition by flowing nitrogen gas through the photocatalytic reactor in order to obtain zero dissolved oxygen throughout the experiment. The second one was aerated with air to obtain the saturated dissolved oxygen around 8.8 mg/l. The third one was carried out at a very high dissolved oxygen level by flowing pure oxygen gas to acquire the saturated dissolved oxygen around 37.7 mg/l.

Fig. 8 shows the effect of dissolved oxygen on the degradation of 2-propanol with and without the UV illumination. The results show that the photocatalytic degradation of 2-propanol with the UV illumination was higher than the system without the UV illumination for both dissolved oxygen levels, 8.8 and 37.7 mg/l. In the absence of the UV illumination, a decrease in the 2-propanol concentration with time was observed, and the removal rate did not depend on dissolved oxygen level. It is understandable that 2-propanol can be easily vaporized from solution during aeration, known as stripping effect.

Under the UV illumination, 2-propanol was degraded rapidly and completely within 1.5 h when the 2-propanol solution contained very high dissolved oxygen of 37.7 mg/l. Interestingly, the system having a high dissolved oxygen level (37.7 or 8.8 mg/l) gave a much higher degradation rate of 2-propanol than that having zero dissolved oxygen. From the present results, it can

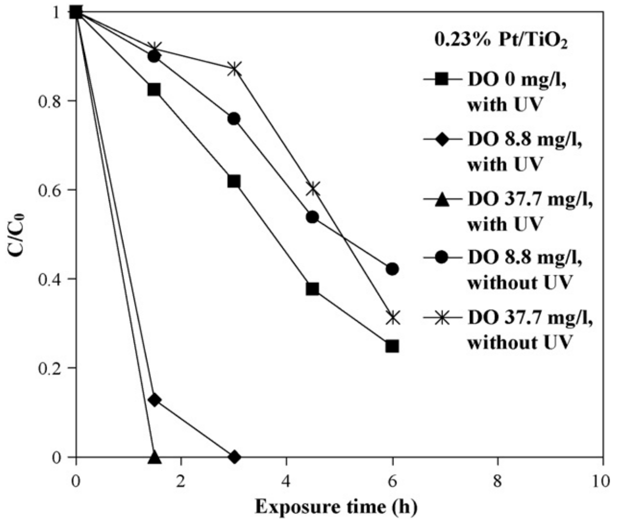

Fig. 8. 2-Propanol concentration ratio as a function of exposure time under different initial dissolved oxygen levels (catalyst dosage, 0.5 g/l; initial 2-propanol concentration, 0.65 mM; initial solution pH, 7; temperature, 25 °C).

be concluded that the dissolved oxygen has a significant role in the photocatalytic degradation of 2-propanol. This is because oxygen molecules act as an electron scavenger to trap and separate electron ($e^-$) out from the positive hole ($h^+$), as shown by Eq. (1), which helps reduce the chance of electron–hole pair recombination. Hence, the photocatalytic process can proceed effectively [23,24].

$$e^- + O_2 \rightarrow {}^{\bullet}O_2{}^- \tag{1}$$

Moreover, oxygen molecules can also be transformed on the surface of Pt to be atomic oxygen, which is a strong oxidant, so it can react with the reaction substrate directly to enhance the overall reaction rate. Furthermore, the oxygen as well as the photogenerated positive hole are also a starting species to produce a very strong oxidant, hydroxyl radical ($OH^{\bullet}$) under light irradiation, as illustrated by Eqs. (2)–(5). Hydroxyl radical is a species that has a very high oxidation potential and can react with organic molecules rapidly and nonselectively [25,26]. In addition, it is worth noting that in the absence of dissolved oxygen, 2-propanol could be photocatalytically degraded, as shown in Fig. 8, due to the formation of hydroxyl radical from the reaction between the positive holes and water molecules according to Eq. (5) in spite of comparatively less amount than the case of the presence of dissolved oxygen. In addition, since there was no significant change of solution pH (not gradually turning to be more acidic) during the courses of reaction, it is then implied that the hydroxyl radical formation due to the reactions shown by Eqs. (2) and (3) becomes more dominant than that due to Eqs. (4) and (5).

$$2H_2O + {}^{\bullet}O_2{}^- \rightarrow 2H_2O_2 + e^- \tag{2}$$

$$H_2O_2 \rightarrow 2OH^{\bullet} \tag{3}$$

$$h^+ + OH^- \rightarrow OH^{\bullet} \tag{4}$$

$$h^+ + H_2O \rightarrow OH^{\bullet} + H^+ \tag{5}$$

As mentioned above, the degradation rate increased with decreasing solution pH because the adsorption of 2-propanol increases with decreasing solution pH. However, the degradation rate increased obviously when the solution pH increased from 10 to 12. This is because hydroxyl radical can form more easily at a very high pH, as supported by Eq. (4).

### 3.4. Intermediate product

In this work, acetone was solely detected as an intermediate product from the photocatalytic degradation of 2-propanol. The results indicate that 2-propanol is degraded to form acetone, and acetone is further oxidized into carbon dioxide and water, as shown by Eqs. (6) and (7), respectively. Interestingly, the rate of 2-propanol was almost constant, indicating that the degradation rate of 2-propanol follows zero-order kinetics. Further detailed investigations on the oxidation pathways of 2-propanol photodegradation, including acetone and other intermediate products formation, will be carried out and presented in the next contribution.

$$CH_3CHOHCH_3 + 2OH^{\bullet} \rightarrow (CH_3)_2CO + 2H_2O \tag{6}$$

$$\begin{tikzcd}(CH_3)_2CO + OH^{\cdot}/{}^{\cdot}O_2^{-} \arrow[r] \arrow[rr, bend right] & \text{Other intermediates} \arrow[r] & CO_2 + H_2O\end{tikzcd} \tag{7}$$

## 4. Conclusions

The activity of either platinum or titania alone was extremely low in the photocatalytic degradation of 2-propanol, but for platinum loaded on titania, the degradation rate of 2-propanol increased significantly. Titania as a semiconductor generates electron–hole pairs under UV illumination. The Pt nanoparticles on the $TiO_2$ surface are responsible for providing adsorption sites for 2-propanol and reducing the electron–hole pair recombination. From the experimental results, the rate of 2-propanol degradation did not depend on the initial 2-propanol concentration and it could be expressed by a zero-order kinetics equation. An increase in the catalyst dosage resulted in increasing the rate of 2-propanol degradation, since an increase in the active sites is responsible for generating sufficient active species to oxidize 2-propanol. Under the acidic condition, the degradation rate of 2-propanol increased drastically because of better adsorption of 2-propanol onto the surface of catalyst. The rate increased again when the solution pH was around 12 because the hydroxyl radical formation rate increased. The dissolved oxygen was revealed experimentally to be an important factor affecting the photocatalytic degradation of 2-propanol, since it can act as an electron scavenger to prevent the recombination process as well as to be responsible for producing hydroxyl radical, an active oxidant.

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