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## HETEROGENEOUS PHOTODEGRADATION OF ORANGE G UNDER THE INFLUENCE OF ADDED OXIDISING AGENTS USING TiO<sub>2</sub> THIN FILM

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**Abstract:** This study examined the effect of added oxidising agents on the photocatalytic performance of immobilised TiO<sub>2</sub> thin film. Two oxidising agents were used, namely O<sub>2</sub> and H<sub>2</sub>O<sub>2</sub> were used. Orange G (OG) was used as the model compound. The experiments were carried out batch-wise using a 200 ml photoreactor and UV light of wavelength 365 nm. It was observed that the addition of O<sub>2</sub> and the peroxide significantly enhanced the photocatalytic process. Adding of pure O<sub>2</sub> and 1mM H<sub>2</sub>O<sub>2</sub> has increased the degradation of 1 mM OG from 2% to 43% and 29%, respectively. Such addition has also improved the biodegradability (based on BOD) of the dyestuff from 4 mg l<sup>-1</sup> to 24 mg l<sup>-1</sup>. While then increase in O<sub>2</sub> concentration can improve the process, the correct dosage of peroxide required optimisation.

**Keyword:** Photocatalysis, TiO<sub>2</sub>, immobilised TiO<sub>2</sub>, reactive dyestuff

**Abstrak:** Kajian telah dijalankan untuk menyiasat kesan penambahan agen pengoksida ke atas kelakunan pemangkinan-foto saput tipis TiO<sub>2</sub>. Dua agen pengoksida telah digunakan iaitu O<sub>2</sub> dan H<sub>2</sub>O<sub>2</sub>. Bahan pewarna Orange G (OG) telah digunakan sebagai sebatian model. Eksperimen telah dijalankan dalam reactor foto dengan pancaran UV pada panjang gelombang 365 nm. Kajian mendapati bahawa penambahan O<sub>2</sub> dan peroksida telah meningkatkan keupayaan proses pemangkinan-foto. Penambahan O<sub>2</sub> (100%) dan 1 mM H<sub>2</sub>O<sub>2</sub> telah meningkatkan tahap penguraian OG dari 2% kepada masing-masing 43% dan 29%. Penambahan kedua-dua bahan ini juga telah dapat mempertingkatkan tahap biorosotan bahan pewarna. Nilai BOD telah meningkat dari 4 mg l<sup>-1</sup> kepada 24 mg l<sup>-1</sup>. Keberkesanan proses meningkat sejajar dengan peningkatan kepekatan O<sub>2</sub> yang digunakan. Dos peroksida perlu dioptimumkan untuk mendapatkan tahap penguraian yang terbaik.

**Katakunci:** Pemangkinan-foto, TiO<sub>2</sub> pegun, pewarna reaktif, saput tipis, pemangkin heterogen

## 1.0 Introduction

The use of metal oxide semiconductor in the presence of UV light, termed as photocatalysis, has gained wide attention since its initial study in the mid' 70s (Pelizzetti *et al.*, 1990; Legrini *et al.*, 1993; Tennakone *et al.*, 1995). Many catalysts have been tested, but TiO<sub>2</sub> in either the hybrid mixture of rutile (approx. 70%) and anatase form or pure anatase form seems to be the best catalyst for photodegradation of various pollutants in term of efficiency, stability, and cost (Tanaka *et al.*, 1989; Andreozzi, 1999; Gogate and Pandit, 2004). It is capable of absorbing UV light up to about 400 nm wavelength for the initiation of the photocatalysis process (Bauer, 1994; Goslich *et al.*, 1997).

Pelizzetti and Minero (1993) has provided a detail discussion on the mechanism of the TiO<sub>2</sub> photocatalysis process. The photocatalysis process is initiated by the absorption of UV radiation by the semiconductor. With the photons' energy equal or greater than the energy of the semiconductor band gap, electrons are promoted from the valence band to the conduction band forming electron-hole pairs (Eqn. 1). The electrons then reduce the dissolved oxygen (if present) forming superoxide radical ions, O<sub>2</sub><sup>-•</sup>, whereas the remaining holes are capable of oxidising adsorbed H<sub>2</sub>O or OH<sup>-</sup> to reactive HO<sup>•</sup> (Eqns. (2) to (4)). Unfortunately, when there are not enough electron acceptors available in water, considerable numbers of electron-hole pairs will recombine, and thus reduce the efficiency of the process.



The concentration, surface area and the nature of the catalyst play an important role in deciding the overall rate of degradation (Thompson, 1997; Goslich *et al.*, 1997; Kumar *et al.*, 1999, Gogate and Pandit, 2004). Additionally, characteristics of the wastewater, pH, temperature, O<sub>2</sub> concentration and the presence of H<sub>2</sub>O<sub>2</sub> have significant effects on the efficiency of the photocatalysis process (Tanaka *et al.*, 2000; Bakar and Aris, 2000).

In a laboratory-scale study, the photocatalysis process is normally carried out in a reactor using slurry of fine solid semiconductor particles dispersed in a liquid phase as the photocatalyst. However, due to the catalyst-solution separation problem (at the end of the process) and fouling at the surface of the radiation source due to catalyst particle decomposition, application of large scale slurry reactors may be problematic. The use of the catalyst in an immobilised form has been made as an alternative to the

slurry with considerable efficiency improvement compare to the slurry form (Hofstadler *et al.*, 1994; Yamashita *et al.*, 1996; Goslich *et al.*, 1997; Freudenhammer *et al.*, 1997; Bauer *et al.*, 1999). Full-scale commercial versions of immobilised catalytic reactors have been reported (Gogate and Pandit, 2004).

In this study, the effectiveness of immobilised TiO<sub>2</sub> photocatalyst in degrading an azo dyestuff, Orange G (OG), was explored. The objective of the study was to explore the possibility of further improving the performance of the process by adding two oxidising agents namely, O<sub>2</sub> and H<sub>2</sub>O<sub>2</sub>.

## 2.0 Methodology

The chemicals used were of analytical grade supplied by Aldrich. The immobilised TiO<sub>2</sub> was prepared using sol-gel dip coating technique (Juahir *et al.*, 1999). After the coating process, approximately 151 mg of TiO<sub>2</sub> of anatase type (Ti(OCH(CH<sub>3</sub>)<sub>2</sub>)<sub>4</sub>) was transparently coated on the surface of a hollow cylindrical pyrex glass (40 mm radius and 130 mm length). Orange G (OG) (Figure 1) with a concentration of 1 mM (453 mg l<sup>-1</sup>) was used in the experiments. A 6 W Sen UVL6DS-12 lamp irradiating at 365 nm was used as a UV source. Oxygen was either supplied by AirSep AS-12-E O<sub>2</sub> generator or Cosmo blower at a rate of 2 l min<sup>-1</sup>, depending on the required O<sub>2</sub> concentration.

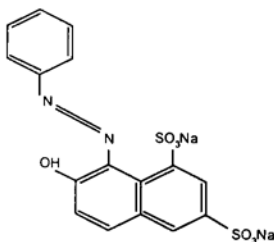


Figure 1: The chemical structure of Orange G

The performance of the process was characterised based on absorbance, biochemical oxygen demand (BOD) and chemical oxygen demand (COD). Absorbance analysis was carried out using UV spectrophotometer (Shimadzu 1601PC) at a wavelength of 475 nm. Analysis of COD and BOD were carried out according to APHA (1992). Due to the refractory nature of the dyestuff, seeding of microorganism had been carried out in the BOD analysis.

Four types of photocatalysis processes were evaluated, UV/TiO<sub>2</sub>/O<sub>2</sub> (21%), UV/TiO<sub>2</sub>/O<sub>2</sub> (100%), UV/TiO<sub>2</sub>/H<sub>2</sub>O<sub>2</sub> (0.1 mM) and UV/TiO<sub>2</sub>/H<sub>2</sub>O<sub>2</sub> (1 mM). A control experiment, UV/TiO<sub>2</sub> was also carried out. Experiments were performed in a cylindrical glass reactor of 200 ml capacity (Figure 2). Mixing in the reactor was provided using a magnetic stirrer. The required concentration of O<sub>2</sub> was achieved by bubbling the solution with either an O<sub>2</sub> generator (for 100% O<sub>2</sub>) or a blower (21% O<sub>2</sub>). The O<sub>2</sub> or air was introduced into the reactor through a sintered glass placed near the bottom of the reactor. The experiments started when the UV lamp was switched on. For the system with H<sub>2</sub>O<sub>2</sub>, the peroxide was added prior to switching on the UV lamp. Samples were then collected at predetermined intervals up to four hours.

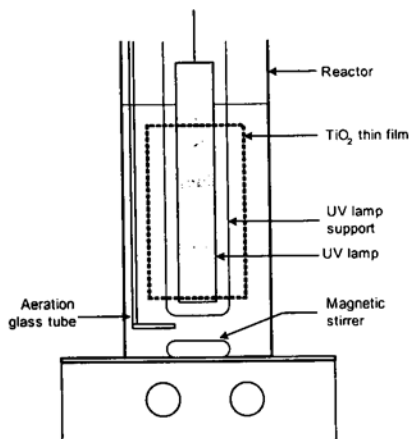


Figure 2: Setup of the photocatalysis reactor

### 3.0 Results and Discussion

#### 3.1 Absorbance

The results of OG degradation based on absorbance is shown in Figure 3. The amount of degradation ranges from about 2% to 43% depending on the type of oxidising agent added into the system. The degradation of OG by the immobilised UV/TiO<sub>2</sub> process was only about 2% after four hours of irradiation. Bubbling the solution with air increased the degradation to about 18% while bubbling the solution with pure O<sub>2</sub> increased the degradation to about 43%. Adding peroxide to the UV/TiO<sub>2</sub> system enhanced the process to about 24% - 29% depending on the concentration of the peroxide. It can be observed that by increasing the peroxide from 0.1 mM to 1 mM did not provide a significant improvement to the process. The degradation of the OG

was found to follow a 1<sup>st</sup> order reaction behaviour with respect to absorbance. The reaction rate constant of the tested systems is given in Table 1. It follows the observation shown in Figure 3 with UV/TiO<sub>2</sub>/O<sub>2</sub> (100%) being the most effective process ( $k = 2.1 \times 10^{-3}$ ).

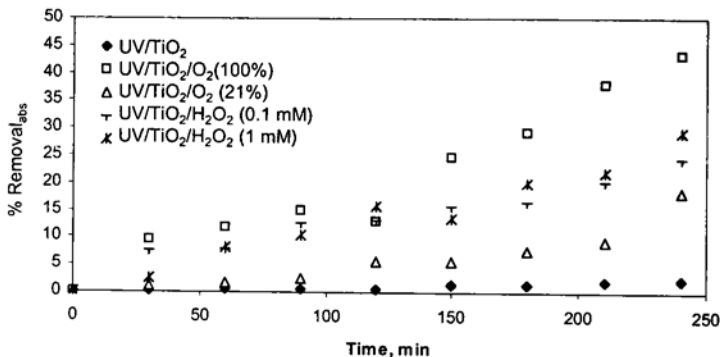


Figure 3: Photocatalytic degradation of Orange G

The degradation of OG in the UV/TiO<sub>2</sub> system is due to the role of HO<sup>•</sup> generated through Eqns. (1) and (2) as explained earlier. In the presence of organic substance, the radicals decompose the organic molecules (RH) generally through hydrogen abstraction forming organic radicals (R<sup>•</sup>) (Eqn. (5)). The organic radicals possess mainly reducing properties which can be consumed through reactions with H<sub>2</sub>O<sub>2</sub> and O<sub>2</sub> (Eqns. (6) and (7)).



As O<sub>2</sub> is needed to complete the oxidation process, the bubbling of pure oxygen is observed to further improve the photodegradation process as compared to normal aeration. Furthermore, O<sub>2</sub> reacts with the electrons in the solution forming O<sub>2</sub><sup>•-</sup> preventing the electron/hole recombination. This improves the oxidation capacity of the process.

Table 1: Reaction rate constant,  $k_1$ , of different immobilised photocatalysis systems

System	Rate constant, $k_1$ ( $\text{min}^{-1}$ )
UV/TiO <sub>2</sub> /O <sub>2</sub> (100%)	$2.1 \times 10^{-3}$
UV/TiO <sub>2</sub> /H <sub>2</sub> O <sub>2</sub> (1 mM)	$1.3 \times 10^{-3}$
UV/TiO <sub>2</sub> /H <sub>2</sub> O <sub>2</sub> (0.1 mM)	$1.2 \times 10^{-3}$
UV/TiO <sub>2</sub> /O <sub>2</sub> (21%)	$6.0 \times 10^{-4}$
UV/TiO <sub>2</sub>	$1.0 \times 10^{-4}$

The use of peroxide as added catalyst although advantageous, apparently needs to be optimised. Similar observations were made by Balcioglu and Arslan (1997). As with O<sub>2</sub>, adding suitable concentration of H<sub>2</sub>O<sub>2</sub> into the UV/TiO<sub>2</sub> process reduces the electron/hole recombination. Besides, it increases the process efficiency through the following reaction.



Excessive dosage of the peroxide inhibits the reaction possibly due to the scavenging effect of H<sub>2</sub>O<sub>2</sub>. The peroxide reacts with the HO<sup>•</sup> to produce HO<sub>2</sub><sup>•</sup> (Eqn. (9)), which is a less reactive species than HO<sup>•</sup>. Furthermore, excessive concentration of H<sub>2</sub>O<sub>2</sub> will also cause a reaction between TiO<sub>2</sub> and the peroxide forming complex molecules such as TiO<sub>2</sub>(OH)<sub>2</sub> and Ti(OOH)(OH)<sub>2</sub> which reduce the photocatalytic activities of the process (Rivera *et al.*, 1999).



### 3.2 Biochemical Oxygen Demand (BOD)

In chemical oxidation process, the BOD of a treated solution is expected to increase in the early stage of the reaction before it starts to decline (Marco *et al.*, 1997). The rate and extent of the rise and decline of the BOD depend on the types of oxidising agent and the treated organics. The increase in BOD value suggests the breakdown of the refractory parent compound into byproducts which are more emendable to microorganism. This is a favourable outcome of the oxidation reaction if the process is to be used as a preliminary treatment prior to biological process. In this case, an optimal chemical oxidation time plays an important role in the cost saving of the integrated process. The optimum time is normally identified corresponding to the peak of the BOD value profile. However, if the process is aimed at mineralising the organic compound, the reaction time needs to be prolonged beyond the peak.

The effect of the added oxidising agents on the BOD of the OG is given in Figure 4. As expected, the BOD value increased with reaction time. The highest BOD value after four hours of reaction was produced by UV/TiO<sub>2</sub>/H<sub>2</sub>O<sub>2</sub> (1 mM). This is followed by UV/TiO<sub>2</sub>/O<sub>2</sub> (100%), UV/TiO<sub>2</sub>/O<sub>2</sub> (21%), UV/TiO<sub>2</sub>/H<sub>2</sub>O<sub>2</sub> (0.1 mM) and UV/TiO<sub>2</sub>. The initial BOD value of OG was about 4 mg l<sup>-1</sup> while the treated BOD value ranged from about 13 mg l<sup>-1</sup> to 24 mg l<sup>-1</sup>. The optimal value of the BOD profile was not observed in this study suggesting that a for longer reaction time is needed.

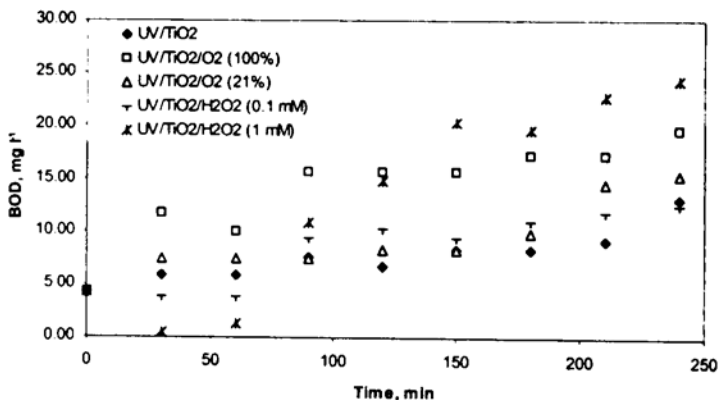


Figure 4: BOD profile for the tested photocatalysis processes

### 3.3 Chemical Oxygen Demand (COD)

The results of the COD value is shown in Figure 5. The removal of COD ranged from 27% to 30%. The highest COD removal was achieved by using UV/TiO<sub>2</sub>/O<sub>2</sub> (100%), followed by UV/TiO<sub>2</sub>/O<sub>2</sub> (21%) and UV/TiO<sub>2</sub>. However, the difference in the COD removal was rather insignificant. It seems that the HO<sup>•</sup> generated by the photocatalytic process was insufficient to break down the byproduct of the oxidation further. It is also possible that the byproducts are quite stable and resistant to HO<sup>•</sup> as compared to the parent compound.

The removal of COD by the UV/TiO<sub>2</sub>/H<sub>2</sub>O<sub>2</sub> process was the lowest. It is possible that the COD value for the sample from this process was affected by the remaining H<sub>2</sub>O<sub>2</sub> in the solution (Kuo, 1992).

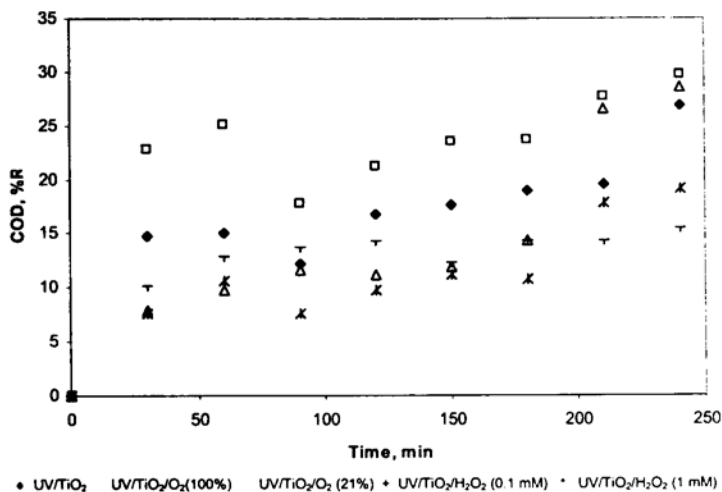


Figure 5: COD removal by the photocatalysis processes

#### 4.0 Conclusions

The effectiveness of the immobilised TiO<sub>2</sub> photocatalyst in degrading a reactive dyestuff, OG, was explored. In particular, the effect of O<sub>2</sub> and H<sub>2</sub>O<sub>2</sub> in improving the process was characterised based on absorbance, BOD and COD. It was found that the immobilised UV/TiO<sub>2</sub> process was not effective in degrading 1 mM of OG, as the degradation (based on absorbance) was only 2%. The effectiveness of the process however improved with the addition of H<sub>2</sub>O<sub>2</sub> or O<sub>2</sub> as co-oxidant. The photocatalytic performance of TiO<sub>2</sub> was improved to 43% with pure O<sub>2</sub> as compared to 29% with the peroxide. The biodegradability of the dye was also improved by the processes while mineralisation requires longer reaction time. In short, while immobilised photocatalyst seems to be a viable alternative to the slurry catalyst, more studies are needed to improve the process to make it acceptable in industrial application.

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where,  $I_s$  is point load strength index;  $B_s$  is block size index;  $W$  is weathering index and  $J_s$  is relative ground structure index. Equation (3), however, does not consider interactions between type and size of machine required.

Rucker (1999) studied rippability of decomposed and slightly weathered medium-coarse granite in Arizona for a highway project. He found that RQD is the best indirect indicator of block size in rock mass for fractal geometry. Ripping causes the fractal structure of the fractured rock mass to self organize into a critical system to resist ripping forces.

More recently, Hakan (2004) conducted a study on surface coal mine in Turkey. He used five parameters, i.e. uniaxial compressive strength (UCS), point load index, seismic velocity, Schmidt hammer value and average discontinuity spacing to assess the excavability. He also proposed the use of specific energy (energy to remove unit volume of material) for the assessments. These parameters are then divided into five main classes with respect to their rippability classification. In addition, Hakan (2004) proposed correlation between specific energy and ripper production, thus allowing this system to predict the production rate with the different dozer sizes.

#### 4.0 Conclusions

Even though many excavability assessments use seismic velocity as an indicator, this method may not provide satisfactory estimates for excavation. These are attributed to geological features, which require different field procedures to be identified. Furthermore the basic material characteristics such as rock type and abrasiveness that affect rippability performance are not represented in seismic velocity. In general, seismic velocity cannot be determined to an accuracy better than 20% or variance of 1000 m/s for similar materials (Kirsten, 1982).

The graphical based method which is simpler and considers only two parameters, i.e. point load strength and fracture spacing indices was then developed. The excavability of rock mass also depends on factors such as joint continuity, gouge, joint set number and direction of discontinuities.

Although the grading system is the most comprehensive assessment method, factors such as moisture content, rock mass properties, topography, bedding thickness and infilled material are not included. Except for MacGregor et al (1994) who addressed the influence of rock type to rippability, others grading assessments are being generalised by type of geological parameters. It should be noted that each rock type displays significant differences in the structural and mode of existence. Igneous rocks, for example, can have many occurrences of boulders, which may have similar parameters, but the size would matter. Normal digging could excavate small boulders easily, but the bigger size would need different technique of excavation. These boulders may cause significant problems during excavation and normally need to be blasted down to a more manageable size. MacGregor et al (1994) found that weathering is a significant variable for igneous rocks

compared to other type of rocks. Table 1 summarises the parameters being used by various researchers. Even though uniaxial compressive strength (UCS) is the most popular parameter, the need for employing simple and practical *in-situ* testing such as point load test and Schmidt hammer are vital. With practical *in-situ* testing, logistic and sampling problems can be minimised.

In sedimentary rocks, the occurrences of bedding, folding, foliation and the multi layer of rock types are few distinctive differences compared to igneous rocks. Shale, which is interbedded with sandstone, would have lower mass strength compared to the sandstone layers. Thus, shale can be excavated by different kind of excavation techniques. However, as the rock mass is always interbedded with low and high strength rocks, the appropriate excavation method could differ from the assessment method. The varying scale of discontinuity that is always present in the sedimentary rock such as thickness of bedding, joints and foliation are not specified in most assessment systems but could significantly influent the ease of excavation. Therefore, consideration on the local inhomogeneity of rocks in the assessment is also important.

The mode of occurrence of rock mass is another important factor in deciding the excavation method. The material might be rippable but the topography would not allow such method to be effective. For the same lithology, the ease of excavating a highly moisturised rock could be easier compared to dried ones.

Despite the number of excavation assessments, they are not designed to deal with tropical weathered rock where inhomogeneity of lithology, moisture content, penetration and the topography make excavatability assessment more complicated. Thus, research effort must be directed to improve the assessment of ripping performance especially for sedimentary rock mass in tropical climate.

Table 1: Summary of parameters considered for excavation assessment

Parameters/References	Strength					Joint/Discontinuity												
	SV	GS	UCS	PLT	SH	TS	ROQ	NJS	VJC	JR	JA	JO	JS	JC	JG	BS	A	W
Caterpillar Tractor Company (2001)	X																	
Atkinson (1971)	X		X	X								X						X
Franklin et al (1971)	X										X				X			X
Bailey (1975)	X		X									X						X
Weaver (1975)	X											X						X
Church (1981)	X		X								X							X
Kirsten (1982)	X		X				X	X	X	X	X	X				X		X
Muftuoglu (1983)	X		X									X						X
Abdul Latif et al (1983)	X		X								X	X	X	X				X
Smith (1986)	X																	X
Komatsu (1987)	X		X									X					X	X
Singh et al (1987)	X		X									X						X
Bozdog (1988)	X		X									X						X
Karpuz (1990)	X		X									X				X		X
MacGregor et al (1994)	X		X									X						X
Pettifer and Fookes (1994)	X		X								X							X
Kramadhrata (1998)	X		X								X							X
Hadjigeorgiou and Poulton (1998)	X		X								X							X
Rucker (1999)	X		X								X							X
Hakan (2004)	X		X								X							X

Note SV= seismic velocity; UCS= uniaxial compressive strength; GS= Grain size; PLT= point load test; SH= schmidt hammer; TS= tensile strength; RQD= rock quality designation; A= abrasiveness; W= weathering; NJS= no. of joint sets; VJC= volumetric joint count; JR= joint roughness; JA= joint alteration; JS= joint spacing; JC= joint continuity; JG= joint gouge; BS= bedding spacing

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