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Effect of inorganic oxidants in photodecolourization of an azo dye

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Abstract

Aqueous solutions of the dye Acid Orange 7 were decolourized in the presence of ultraviolet irradiation (UV) and photocatalyst, TiO_2. Decolourization rate was increased in the presence of UV/TiO_2 compared to UV alone. Addition of hydrogen peroxide, H_2O_2, in concentrations ranging from 8.82×10^{-3} to 7.1×10^{-2} M to the solution containing pollutant and TiO_2 (0.5 g l^{-1}) resulted in increased rates of decolourization. The rate of dye decolourization using periodate, $NaIO_4$, (4.76×10^{-5} to 1×10^{-3} M) at TiO_2 concentration of 0.5 g l^{-1} under UV illumination was higher than that for UV/TiO_2/H_2O_2 and increases with periodate concentration. The rate of decolourization of Acid Orange 7 was found to follow first-order kinetics. Reaction rate orders with respect to the oxidant species were 0.51 and 0.8, respectively for H_2O_2 and $NaIO_4$. Kinetic parameters of the photodecolourization process such as apparent rate constant (k_{app}) and initial reaction rate ($R_{initial}$) and efficiency parameters such as apparent quantum yield (Q_{app}) and electrical energy per order (EE/O) were estimated and compared for UV/TiO_2, UV/TiO_2/H_2O_2 and UV/TiO_2/IO_4^- catalytic systems.
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Keywords: Decolourization; Dyes; Titanium dioxide; Hydrogen peroxide; Sodium periodate; Apparent quantum yield; Electrical energy per order

1. Introduction

Dyeing and finishing of textile goods is a major concern to the environmentalist, due to the introduction of large quantities of colour, chemical oxygen demand, nonbiodegradable organics, and other hazardous chemicals into the process effluents [1,2]. Whilst most dyes are not particularly toxic, the presence of dyes in surface waters is an obvious indicator of environmental pollution. Advanced oxidation processes (AOPs) represent an alternative method of removing organic pollutants from water which does not entail any of the disadvantages of the adsorptive technologies. Whereas a small number of semiconductor materials has been identified as being applicable for this purpose, in practice the majority of studies in this area have employed the anatase form of TiO_2 [3]. All AOPs have as a common basis the generation of free radicals. The powerfully oxidizing hydroxyl radical ($^{\bullet}OH$) is present in all AOPs, but other radical species may also be involved. These free radicals may be produced by a number of different ways. This includes the use of oxidants such as hydrogen peroxide or ozone with ultraviolet light (UV) [4–6].

This work involves the use of an AOPs to decolourize the azo dye Acid Orange 7. The effectiveness of the three systems UV/TiO_2, UV/TiO_2/H_2O_2 and UV/TiO_2/IO_4^- under UV irradiation on the decolourization of aqueous solutions of Acid Orange 7 in a laboratory-scale photoreactor will be compared. The efficiency and kinetic parameters of the AOPs will be quantified and compared for the three catalytic systems used.

2. Experimental procedures

2.1. Reagents

TiO_2 (Degussa P25, average particle size about 30 nm, 70% anatase form and surface area about 50 m^2 g^{-1}) was purchased from Aldrich. Acid Orange 7 shown in Fig. 1 was purchased from Fluka. All other chemicals [sodium periodate, hydrogen peroxide (30%, w/v), and alkali for pH adjustment] were obtained from Fisher Scientific.

2.2. Irradiation experiments

Aqueous solutions of Acid Orange 7 (5×10^{-5} M) were prepared in double distilled water. The pH of the TiO_2/dye suspension was 6.1. Addition of H_2O_2 or IO_4^- to the TiO_2/dye

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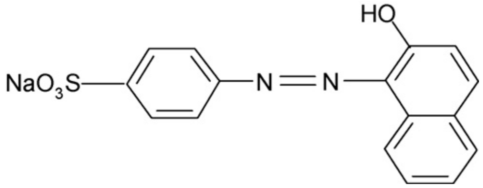

Fig. 1. Structure of azo dye Acid Orange 7.

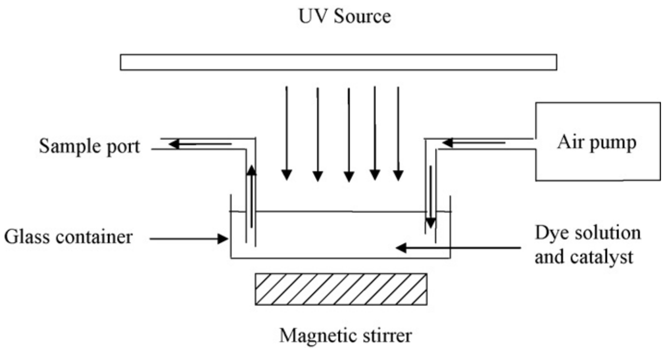

Fig. 2. Schematic diagram of the batch photoreactor.

suspension decreases the pH below 6.1. A few drops of an alkali were added to adjust the pH to its original value (6.1). The reactor used for studying the photodegradation of the dye in all experiments is a batch photoreactor (Fig. 2). It is consists of a glass container (1000 ml). The contents (TiO_2/dye or TiO_2/dye/oxidant) of the container were agitated by a magnetic stirrer and kept purged with air at a rate of 3000 ml min^{-1}. Irradiation was carried out with a tubular low pressure mercury lamp immediately after addition of TiO_2 to the dye solution (total rating 43 W, total output at 254 nm 13.4 W, and length 40 cm, Voltarc Tubes Inc., USA) which was located 10 cm from the surface of the dye solution. The total intensity reaching the slurry solution was measured using a UVX radiometer (UV Products Ltd., Cambridge) equipped with a sensor with peak sensitivity at 254 nm was 4 mW cm^{-2}. The local volumetric rate of energy reaching the solution was 2.1 μeinstein s^{-1} l^{-1} at 254 nm. The colour fading of the dye was analyzed spectrophotometrically at its maximum absorption wavelength of 483 nm, using a Shimadzu Model 1601PC double beam spectrophotometer. Samples containing TiO_2 powder were taken periodically from the photoreactor and measured after filtration using 0.2 μm polyethersulfone membrane.

3. Results and discussion

3.1. *Effect of addition of H_2O_2*

The effect of addition of hydrogen peroxide at different concentrations (8.82×10^{-3} to 7.1×10^{-2} M) to TiO_2 (0.5 g l^{-1}) for decolourization of Acid Orange 7 under UV irradiation is shown in Fig. 3. The data plotted according to a first-order model for dye destruction. UV alone in absence of TiO_2 and hydrogen peroxide was found to have no measurable effect on the decolourization of Acid Orange 7 over the longest time employed in the experiment. The rate of dye decolourization increased rapidly with UV/TiO_2 compared to UV only due to the formation of hydroxyl radicals which are the primary oxidizing species due to their highly electrophilic character [6]. It was found that addition of hydrogen peroxide (8.82×10^{-3} M) to TiO_2 (0.5 g l^{-1}) under UV irradiation enhances the decolourization rate of the dye over UV/TiO_2. Increasing the concentration of peroxide to (7.1×10^{-2} M) resulted in increasing the rate of decolourization.Values of the first-order rate constant, k_{app}, were obtained from slopes of the lines of plots of $\ln(C/C_0)$ against time where C_0 represented the initial Acid Orange 7 concentration and C is the dye concentration at time t. The k_{app} ranged from 5.3×10^{-2} to 15.44×10^{-2} min^{-1} (Table 1). The effect of rate determining species (i.e H_2O_2) is expressed by a power law relation:

$$\mathrm{k_{app}} = k[H_2O_2]^n \qquad (1)$$

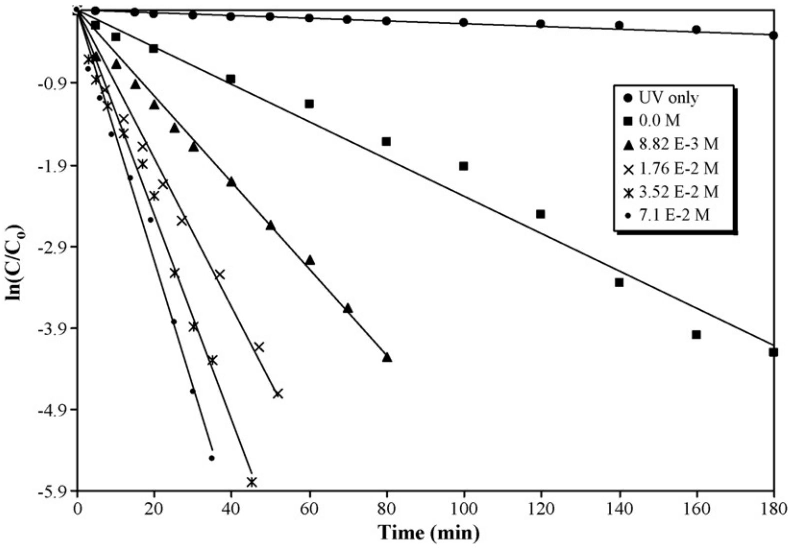

Fig. 3. First-order model for UV induced decolourization of Acid Orange 7 in the presence of TiO_2 (0.5 g l^{-1}) and different concentrations of hydrogen peroxide (M).

Table 1
Apparent rate constants, half life times, and reaction orders for degradation of Acid Orange 7

Photocatalytic system	Concentration	k_{app} (min^{-1})	$t_{1/2}$ (min)	Apparent reaction order (n)
UV	–	0.16×10^{-2}	433.13	–
UV/TiO_2	$0.5\,g\,l^{-1}$	2.28×10^{-2}	30.39	–
UV/TiO_2(0.5 g l^{-1})/H_2O_2	8.82×10^{-3} (M)	5.30×10^{-2}	13.07	0.51
	1.76×10^{-2} (M)	9.12×10^{-2}	7.59	
	3.52×10^{-2} (M)	12.62×10^{-2}	5.49	
	7.10×10^{-2} (M)	15.44×10^{-2}	4.49	
UV/TiO_2(0.5 g l^{-1})/IO_4^-	4.67×10^{-5} (M)	6.1×10^{-2}	11.36	0.80
	1×10^{-4} (M)	17.51×10^{-2}	3.96	
	4.67×10^{-4} (M)	52.22×10^{-2}	1.33	
	1×10^{-3} (M)	74.74×10^{-2}	0.93	

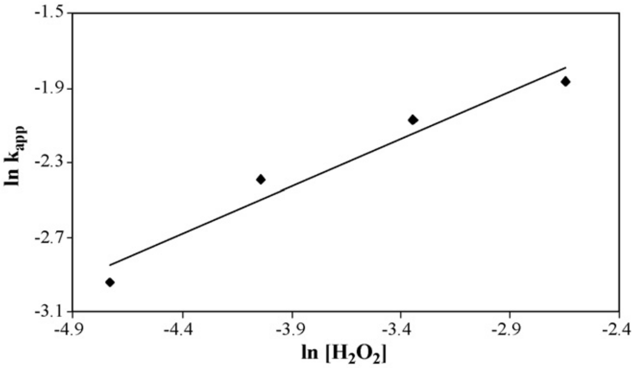

Fig. 4. The influence of hydrogen peroxide concentration on the apparent first-order rate constant.

where k_{app} and k are the apparent and the true rate constants. The exponent, n, the order of reaction with respect to the oxidant species, was obtained by plotting the logarithm of k_{app} againest the logarithm of the hydrogen peroxide concentration (Fig. 4), which yielded a value for n of 0.51 (Table 1).

The enhancement of the rate of dye decolourization using UV/TiO_2/H_2O_2 system may be attributed to the trapping of the photogenerated conduction band electrons of TiO_2 by H_2O_2, which is more efficient than trapping by O_2, according to the following reaction [7,8]:

$$H_2O_2 + e^-_{(cb)} \rightarrow {}^-OH + {}^\bullet OH \qquad (2)$$

In addition to the scavenging action, which inhibits the recombination reaction between the photogenerated carriers (electrons and holes), reaction (2) generates other oxidizing species ($^\bullet$OH), which can participate to the overall oxidative degradation process. The principal output of the used UV source was at 254 nm, so that substantial photolysis of the H_2O_2 would have occurred producing more hydroxyl radicals [9,10]:

$$H_2O_2 \xrightarrow{hv} 2{}^\bullet OH \qquad (3)$$

Increasing H_2O_2 concentration increases both electron scavenging action, and hydroxyl radicals which oxidize the dye and lead to a higher rate of degradation.

3.2. Effect of addition of IO_4^-

The decolourization of Acid Orange 7 irradiated with UV using TiO_2 (0.5 g l^{-1}) at different concentrations of periodate (4.67×10^{-5} to 1×10^{-3} M) is shown in Fig. 5. Photodegrada-

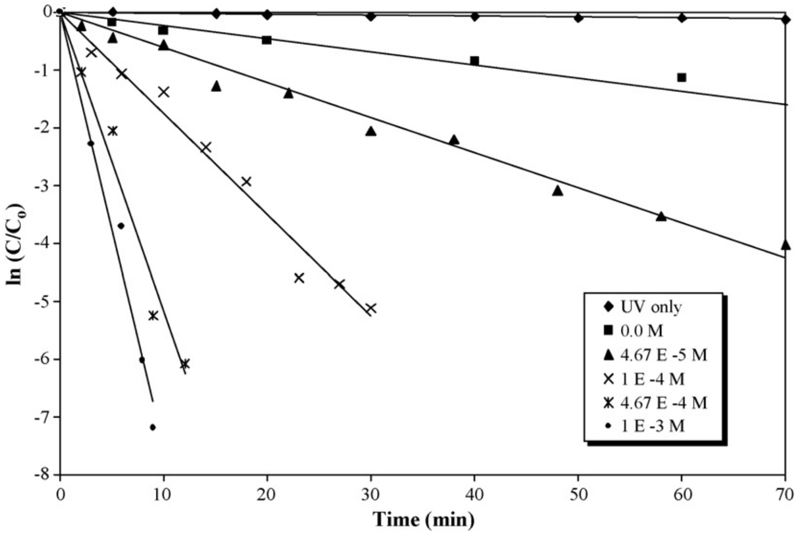

Fig. 5. First-order model for UV induced decolourization of Acid Orange 7 in the presence of TiO_2 (0.5 g l^{-1}) and different concentrations of sodium periodate (M).

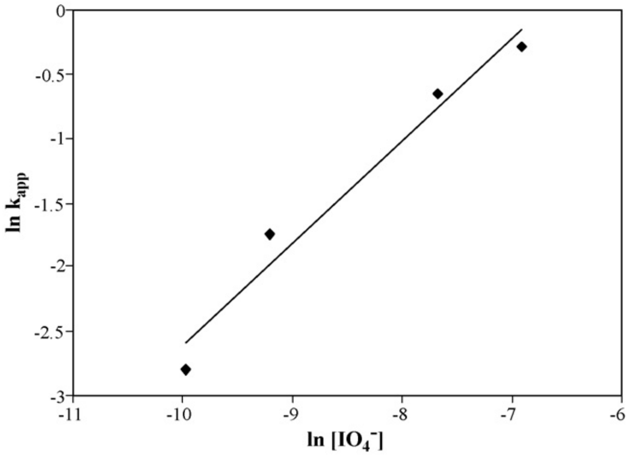

Fig. 6. The influence of sodium periodate concentration on the apparent first-order rate constant.

tion follows first-order model with respect to the dye. The rate of dye decolourization using $UV/TiO_2/IO_4^-$ is higher than that for UV/TiO_2 and increases with increasing periodate concentration from 4.67×10^{-5} to 1×10^{-3} M. The apparent rate constants (6.1×10^{-2} to 74.74×10^{-2} min^{-1}) were greater than those for peroxide (Table 1). The reaction rate order with respect to periodate, obtained from Fig. 6, was found to be 0.8, higher than that for peroxide.

IO_4^- increases the decolourization of Acid Orange 7 by capturing the electrons ejected from TiO_2 so that the probability of recombination of electrons and holes will decrease, i.e. the available number and the survival time of holes will be higher and thus react effectively with adsorbed water molecules to produce more hydroxyl radicals. IO_4^- is one group of oxidants used in UV/TiO_2/oxidant system which has more than two atoms of oxygen and one atom of halogen (I) as a central atom. Polarizability difference in oxidant atoms makes its central atom extremely electropositive. Therefore, IO_4^- can capture the electrons ejected from TiO_2 photocatalyst more than other oxidants [11–13].

$$IO_4^- + 8e_{(cb)}^- + 8H^+ \rightarrow 4H_2O + I^- \tag{4}$$

Also the photolytic decomposition of periodate under UV irradiation (254 nm) involves the formation of a number of highly reactive radical- and non-radical intermediates as follows [2,5,14]:

$$IO_4^- + h\nu \rightarrow IO_3^{\bullet} + O^{\bullet -} \tag{5}$$

$$O^{\bullet -} + H^+ \leftrightarrow {}^{\bullet}OH \tag{6}$$

$$^{\bullet}OH + IO_4^- \rightarrow {}^{-}OH + IO_4^{\bullet} \tag{7}$$

$$H_4IO_6^- + h\nu \rightarrow H_3IO_5^{\bullet -} + {}^{\bullet}OH \tag{8}$$

$$H_3IO_5^{\bullet -} \rightarrow IO_3^- + H_2O + {}^{\bullet}OH \tag{9}$$

These intermediates contribute to the dye degradation as follows: it is known that the colour of the azo dyes such as Acid Orange 7 is due to the presence of the azo bonds (–N=N–). The radical intermediates (with highly electrophilic character) produced from photolysis of IO_4^- attack the azo groups of the dye molecules causing oxidative cleavage. Therefore partial decolourization of the dye has occurred. Increasing the concentration of IO_4^- leads to an increase in the number of radicals formed, so higher decolourization of the dye was achieved in a short time. Degradation of all azo groups will lead to complete decolourization of the dye [13,15,16].

Wang and Hong [5] found that the addition of 10^{-2} M of KIO_4, $K_2S_2O_8$, or H_2O_2 into the UV/TiO_2 system with a TiO_2 concentration of 25 mg l^{-1} resulted in a much faster degradation rate of 2-chlorobiphenyl compared to that in the UV/TiO_2 system. The most effective oxidant for the degradation is found to be KIO_4. Also, the photodecolourization of neutral red dye by UV/ZnO, $UV/ZnO/S_2O_8^{2-}$ and $UV/ZnO/IO_4^-$ systems was reported by Sadik and his co-workers [17]. They found that the decolourization rate follows the order: $UV/ZnO/IO_4^- > UV/ZnO/S_2O_8^{2-} > UV/ZnO$. Saquib and Muneer [18] studied the photocatalytic degradation of Acid Orange 8 dye in aqueous suspensions in the presence of inorganic oxidants (H_2O_2 or $KBrO_3$). The degradation rate of the dye was enhanced and H_2O_2 was found to be the more effective oxidant. Also Saquib and Muneer [19] found that the addition of H_2O_2 or $(NH_4)_2\,S_2O_8$ enhanced the degradation rate of gentian violet in the presence of UV and TiO_2. They found that $(NH_4)_2\,S_2O_8$ had a greater effect than H_2O_2. In accordance with our results presented here for Acid Orange 7, all the work studied in literatures shows that the $UV/TiO_2/IO_4^-$ had a greater effect than $UV/TiO_2/H_2O_2$ or UV/TiO_2 in photodegradation of dyes.

The half life time ($t_{1/2}$) of the first-order reaction is the time required for the reactants to be degraded to half of their initial concentrations. The relationship between $t_{1/2}$ and k_{app} is given by:

$$t_{1/2} = \frac{0.693}{k_{app}} \tag{10}$$

Also the initial reaction rate for the first-order reaction ($R_{initial}$) is related to k_{app} by the following equation:

$$R_{initial} = C_0 k_{app} \tag{11}$$

where C_0 is the initial dye concentration.

It was found that $t_{1/2}$ decreases with increasing the concentration of the inorganic oxidant (hydrogen peroxide or periodate), while the initial reaction rate increases (Tables 1 and 2).

3.3. Efficiency of the reactions

The efficiency of the heterogeneous photocatalytic process can be quantified in terms of quantum yield, which may be defined as the rate at which reactant molecules disappear or product molecules are formed, divided by the number of photons absorbed per unit time. In heterogeneous photocatalysis, the species absorbing light is the photocatalyst such as TiO_2, which may be dispersed as a slurry in an aqueous medium, or may be coated on a solid support in contact with the reactants. With such dispersed or coated catalysts, a large fraction of the incident

Table 2
Initial reaction rate, electrical energy per order, and apparent quantum yield for degradation of Acid Orange 7

Photocatalytic system	Concentration	R_{initial} (mol l^{-1} min^{-1})	EE/O (kWh)	Q_{app}, % (μmol/μeinstein)
UV	–	0.08×10^{-6}	3903.60	0.0635
UV/TiO_2	0.5 g l^{-1}	1.14×10^{-6}	273.93	0.90
UV/TiO_2(0.5 g l^{-1})/H_2O_2	8.82×10^{-3} (M)	2.65×10^{-6}	117.84	2.10
	1.76×10^{-2} (M)	4.56×10^{-6}	68.48	3.62
	3.52×10^{-2} (M)	6.31×10^{-6}	49.49	5.00
	7.10×10^{-2} (M)	7.72×10^{-6}	40.45	6.13
UV/TiO_2(0.5 g l^{-1})/IO_4^-	4.67×10^{-5} (M)	3.05×10^{-6}	102.39	2.42
	1×10^{-4} (M)	8.76×10^{-6}	35.67	6.95
	4.67×10^{-4} (M)	26.1×10^{-6}	11.97	20.72
	1×10^{-3} (M)	37.37×10^{-6}	8.36	29.66

light is either reflected or scattered. There does not usually exist any possibility to determine experimentally the amount of light absorbed by the photocatalyst. In order to bypass the difficulty of determining quantum yields in heterogeneous photocatalysis, another parameter often reported is the apparent quantum yield defined as [20,21]:

$$\text{apparent quantum yield } (Q_{\text{app}}, \text{mol/einstein}) = \frac{\text{rate of disappearance of reactant molecules}}{\text{rate of incident photons inside reactor cell}} = \frac{k_{\text{app}}C_0}{I} \tag{12}$$

where k_{app} is the apparent first-order rate constant, C_0 is the initial dye concentration and I is the total intensity of incident photons entering the reactor cell.

It was observed from Table 2 that Q_{app} for UV/TiO_2/IO_4^- system is higher than those for UV/TiO_2/H_2O_2 and UV/TiO_2 systems. This may be due to the higher activity of periodate compared to hydrogen peroxide as explained before [5,11,13].

3.4. Figure of merit

The evaluation of the efficiency of photodegradation treatment processes is difficult because the reaction rate depends on many factors. Thus, there is need for a figure of merit that can be used to assess the relative performance of each system. As a figure of merit, the electrical energy per order (EE/O) has been proposed, defined as the electrical energy (in kWh) required to reduce the concentration of a pollutant by one order of magnitude in 1000 U.S. gallons (3785 l) of water. The EE/O value may be calculated from the following equation [21]:

$$\text{EE/O} = \frac{P \times (t/60) \times 3785}{V \times \log[C_0/C_f]} \tag{13}$$

where P is the lamp power (in kW), t is the irradiation time (in min), V is the reactor volume (in l) and C_0 and C_f are the initial and final concentrations over the irradiation time. The EE/O values are related to the first-order rate constant, k_{app}, (per minute) by:

$$\text{EE/O} = \frac{145.25P}{V \times k_{\text{app}}} \tag{14}$$

The EE/O was found to decrease with increasing the concentration of hydrogen peroxide and periodate. The EE/O (8.36 kWh) is more favorable at [IO_4^-] of 1×10^{-3} M compared to the other concentrations of hydrogen peroxide or periodate (Table 2). It is established that k_{app} is the key parameter determining the efficiency of the degradation of Acid Orange 7, through affecting $t_{1/2}$, R_{initial}, EE/O, and Q_{app}. From equations (10)–(12) and (14) the increase in k_{app} (indicating higher degradation efficiency) will lead to concomitant decrease in $t_{1/2}$, EE/O, and increase in R_{initial} and Q_{app}.

The results of apparent quantum yields obtained by UV, UV/TiO_2, UV/TiO_2/H_2O_2 and UV/TiO_2/IO_4^- advanced oxidation processes are shown in Table 2. Obviously, the obtained quantum yield value is very low using UV only. Combination of UV/TiO_2, UV/TiO_2/H_2O_2 or UV/TiO_2/IO_4^- shows higher degradation efficiency of the dye and consequently higher values of the quantum yield and lower values of electrical energy per order. The degradation efficiency of these systems could be arranged in ascending order as follows:

$$\text{UV} < \text{UV/TiO}_2 < \text{UV/TiO}_2/\text{H}_2\text{O}_2 < \text{UV/TiO}_2/\text{IO}_4^-$$

The apparent rate constant (k_{app}) of the dye degradation obtained by the four catalytic systems mentioned previously is shown in Fig. 7.

Daneshvar et al. [22] studied the photooxidative decolourization of four textile dyestuffs, C.I. Acid Orange 7 (AO7), C.I. Acid Orange 8 (AO8), C.I. Acid Orange 52 (AO52) and C.I. Acid Blue 74 (AB74), by UV/H_2O_2 in a laboratory scale photoreactor. It was found that the figure-of-merit electrical energy per order (EE/O) is appropriate for estimating the electrical energy efficiency. The EE/O values were found to depend on the concentration of H_2O_2, concentration and basic structure of the dye.

The decolouring and the degradation of xylenol orange in aqueous solution photocatalyzed by band-gap-irradiated TiO_2, modified with nanosized palladium particles has been studied by Iliev et al. [23]. It was found that the quantum yield of the photodestruction reaction of the studied dye is higher than that of using UV/TiO_2 only.

The degradation of three commercially available textile azo dyes, Solophenyl Green (SG), Erionyl Red B (ER) and Chro-

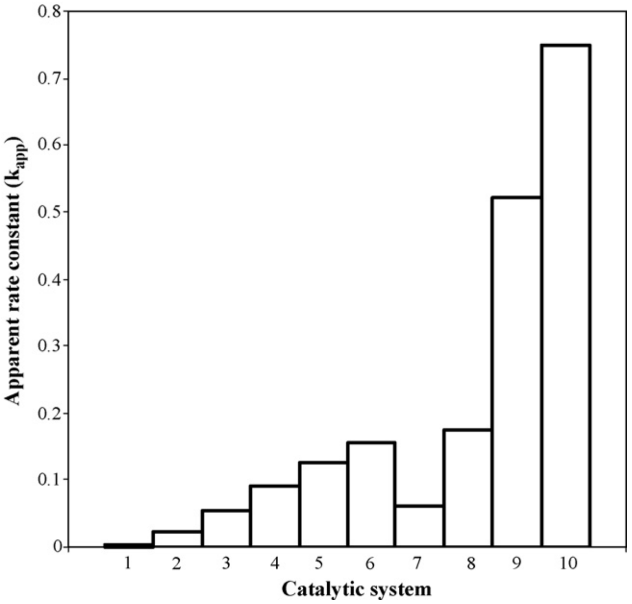

Fig. 7. The apparent rate constant of degradation (k_{app}) of Acid Orange 7 by four different catalytic systems. 1: UV, 2: UV/TiO_2 ($0.5\,g\,l^{-1}$), 3: UV/TiO_2 ($0.5\,g\,l^{-1}$)/H_2O_2 (8.82×10^{-3} M), 4: UV/TiO_2 ($0.5\,g\,l^{-1}$)/H_2O_2 (1.76×10^{-2} M), 5: UV/TiO_2 ($0.5\,g\,l^{-1}$)/H_2O_2 (3.52×10^{-2} M), 6: UV/TiO_2 ($0.5\,g\,l^{-1}$)/H_2O_2 (7.10×10^{-2} M), 7: UV/TiO_2 ($0.5\,g\,l^{-1}$)/IO_4^- (4.67×10^{-5} M), 8: UV/TiO_2 ($0.5\,g\,l^{-1}$)/IO_4^- (1×10^{-4} M), 9: UV/TiO_2 ($0.5\,g\,l^{-1}$)/IO_4^- (4.67×10^{-4} M), 10: UV/TiO_2 ($0.5\,g\,l^{-1}$)/IO_4^- (1×10^{-3} M).

motrope 2R (C2R), has been studied by Faria et al. [24] using photochemical and photocatalytic processes under UV irradiation. The photocatalytic process, using either slurry of Degussa P25 TiO_2 or a biphasic mixture of TiO_2 and activated carbon (AC), more effectively bleaches heavier coloured solutions. A positive effect on the efficiency parameters was observed on the photocatalytic degradation over TiO_2 + AC mixture when compared to TiO_2 alone, which was explained in terms of a synergy induced by the introduction of AC. The efficiency of the different photo-induced degradation processes was evaluated in terms of apparent rate constants, and initial quantum yields. Overall, the results revealed the superiority of photocatalytic processes over neat photolysis and the advantage of using a suspension of TiO_2 + AC mixture instead of TiO_2 alone.

The efficiency of photocatalytic degradation in the previous studies was carried out for different systems such as UV/H_2O_2, UV/TiO_2 or UV/TiO_2 + AC. In our study there was a need to examine the effect of addition of some inorganic oxidants (H_2O_2 and $NaIO_4$) to UV/TiO_2 to detect presence or absence of a synergism between them. In accordance with our results presented here for Acid Orange 7, the values of EE/O and Q_{app} were more effective compared with the previous studies.

4. Conclusions

The Acid Orange 7 can be decolourized by UV/TiO_2, UV/TiO_2/H_2O_2 and UV/TiO_2/IO_4^- systems. It can be seen that UV/TiO_2/IO_4^- was the most effective system at a fixed concentration of TiO_2, $0.5\,g\,l^{-1}$, bringing about high rates of dye decolourization at low periodate concentrations. The rate of decolourization was found to follow first-order kinetics and reaction rate orders with respect to the oxidant species were obtained. The apparent quantum yield (Q_{app}) of the decolourization process was calculated for both UV/TiO_2/H_2O_2 and UV/TiO_2/IO_4^- catalytic systems and was higher for periodate at all concentrations than that for hydrogen peroxide.

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