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Photocatalytic generation of sulphate and hydroxyl radicals using zinc oxide under low-power UV to oxidise phenolic contaminants in wastewater

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ABSTRACT

Photochemical and photocatalytic reaction and the combination of both for the oxidation of phenol in aqueous solution were investigated. Photochemical reaction was carried out using three different types of oxidants in the presence of UV radiation while photocatalytic reaction was carried out on zinc oxide under UV radiation. Three different oxidants viz peroxymonosulphate (PMS), peroxydisulphate (PDS) and hydrogen peroxide (H_2O_2) were employed to generate active sulphate/hydroxyl radicals for oxidation. The synergetic effect of combination of UV/oxidant/ZnO was investigated for the three different oxidant systems. It was found that homogeneous photochemical oxidation of phenol using PMS, PDS or H_2O_2 exhibited much better performance than heterogeneous photocatalytic oxidation using UV/ZnO. The combination of photochemical and photocatalytic oxidation by combining UV/oxidant with ZnO at low UV radiation showed different degradation trends depending on the oxidant. Scattering of UV radiation in UV/PDS/ZnO and UV/H_2O_2/ZnO systems showed a slower rate of phenol degradation as compared to the photochemical oxidation under UV/PDS and UV/H_2O_2 systems. However, UV/PMS/ZnO showed higher rate of phenol oxidation as compared to the photochemical oxidation under UV/PMS, which was mainly attributed to the self-chemical reaction between ZnO and PMS. The phenol degradation in UV/PMS/ZnO followed the first-order kinetics while the phenol degradation resulted from the self-chemical reaction of ZnO/PMS followed zero-order kinetics.

1. Introduction

The pursuit for an efficient and economical technique to destroy the pollutants in wastewater has been an interest of industry and academics. Among the various techniques explored, photocatalytic oxidation has attracted an increased attention in the past few decades [1–4]. Direct oxidation of organics in the presence of UV irradiation (photolysis) has been found to be successful for the treatment of organic-contaminated wastewater. However, this technique faces the disadvantages of high-power requirements and failure to treat refractory compounds. To make the process cost effective, homogeneous photochemical oxidation (in the presence of an oxidant) and heterogeneous photocatalytic oxidation (in the presence of a photocatalyst) were proposed to achieve efficient oxidation under low-power UV radiation.

To date, the anatase form of TiO_2 has been successfully used for photocatalytic treatment of contaminants due to its faster electron transfer when exposed to UV radiation, non-toxicity, and long-term stability [5–11]. As an alternative to TiO_2, zinc oxide, having a similar band gap of 3.2 eV, has also been reported as an effective photocatalyst [12–14]. A few studies have supported the assertion that ZnO is a better photocatalyst than TiO_2, especially for chlorinated compounds using hydroxyl radicals [15]. In order to make photocatalytic oxidation more efficient, the addition of electron scavengers, such as oxygen or hydrogen peroxide, has been utilised. These electron scavengers perform dual tasks of scavenging electrons to improve photocatalytic efficiency and producing active oxygen or peroxide radicals for the photochemical oxidation of the contaminants. The production of active radicals for photochemical oxidation is known to occur either by the chemical reaction of an oxidant with the conduction band electrons of the photocatalyst or by direct absorption of UV radiation. In the past years, photocatalytic oxidation incorporating hydroxyl radicals from H_2O_2 has been investigated extensively. Recent investigations have shown that sulphate radicals exhibit better activity than hydroxyl radicals [16,17]. Sulphate radicals can be generated by the reaction between sulphate-based oxidants (peroxymonosulphate (PMS) and peroxydisulphate (PDS)) and transition metal ions, such as cobalt, iron, and silver [18,19]. Similar to hydrogen peroxide, the sulphate-based

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oxidants show significant photochemical activity by generating active sulphate radicals under the influence of UV radiation. Yang et al. [20] have studied the photochemical degradation of 2,4-dichlorophenol in the presence of PMS and PDS under UV radiation. The combination of TiO_2 based photocatalytic oxidation along with the addition of sulphate-based oxidation as a quenching reagent has been reported [21,22]. However, most previous studies have focused on high-power UV light which tends to favour the combination of photochemical reaction, due to direct UV absorption with the oxidant, and photocatalytic reaction.

In the current study, we compared photochemical oxidation of phenol by UV/oxidant, using PMS, PDS and H_2O_2, and photocatalytic oxidation by UV/ZnO system in the presence of low-power UV light to oxidise phenolic contaminants in water. The synergistic effect of photochemical and photocatalytic oxidation was examined. Experiments were also carried out in the absence of light to understand the direct chemical reaction between ZnO and PMS for phenol oxidation.

2. Experimental

2.1. Reagents

Zinc oxide (ZnO, $Dp = 1–2\ \mu m$, $S_{BET} = 7.5\ m^2/g$) obtained from B.D.H (Australia) was used as received without further treatment. TiO_2, Degussa P25 ($Dp = 15–21$ nm, $S_{BET} = 55.5\ m^2/g$) was obtained from Degussa, Germany. Pure potassium persulphate ($K_2S_2O_8$, PDS), peroxymonosulphate (PMS), and H_2O_2 (30 wt.%) were obtained from Aldrich. Phenol obtained from Aldrich was used to prepare a stock solution at the concentration of 5000 ppm, and the solution was stored in dark until used. Pure methanol (99.5%) was used as a quenching agent to stop the reaction for analysis of phenolic compounds using a HPLC.

2.2. Reactor setup and photodegradation testing

A 1-L reactor with a circular base and a jacket was used in the study. The reactor was placed on a magnetic stirring plate with constant mixing at 400 rpm. The whole system was kept in a laminar flow chamber fitted with a germicidal UV-C lamp of 30 W with the wavelength centred at 253 nm. The reactor position was fixed at 20 cm below the UV lamp in all experiments. In spite of the lamp not being in the axial position with respect to the reactor, preliminary experiments confirmed a positive influence of the UV radiation on the experiment. The intensity of the radiation was measured using a UV probe and a light meter. A syringe was attached to the reactor outlet to withdraw samples at fixed intervals during 6 h runs. In each test, 500 ml of 25 ppm phenol solution was prepared by diluting the stock solution with deionised water. Before the start of the experiment a fixed amount of oxidant was added to the reaction solution and stirred until dissolved. The experiment was started by the addition of a fixed amount of photocatalyst and UV exposure. A sample of 0.5 ml was withdrawn at fixed intervals using a filter syringe. The samples were quenched by adding excess methanol and analysed on a HPLC. A similar procedure was used for dark reactions in the absence of UV light.

3. Results and discussion

3.1. Photolytic and photocatalytic degradation of phenol with PDS

Preliminary experiments were carried out to observe phenol degradation under different reaction conditions. As seen in Fig. 1, a control experiment with only ZnO in solution showed a negligible change in phenol concentration after 5 h, indicating no significant surface adsorption of phenol on the photocatalyst particles. Another control experiment carried out under UV radiation for photolytic decomposition of phenol showed a minor 10% reduction in phenol concentration resulted from the direct absorption of UV radiation by phenol. The experiment with TiO_2 exhibited phenol degradation under UV radiation, however, the removal efficiency was low with only 20% phenol degradation at 5 h. Meanwhile, the reaction carried out in the presence of ZnO under photocatalytic UV irradiation showed a significant degradation of phenol around 55% within 5 h, suggesting high oxidation capacity of the active holes formed on the ZnO catalyst surface induced by UV radiation. Our previous results using ZnO and TiO_2 under high-power UV radiation also showed the similar observation that ZnO exhibited a higher activity than TiO_2 [22].

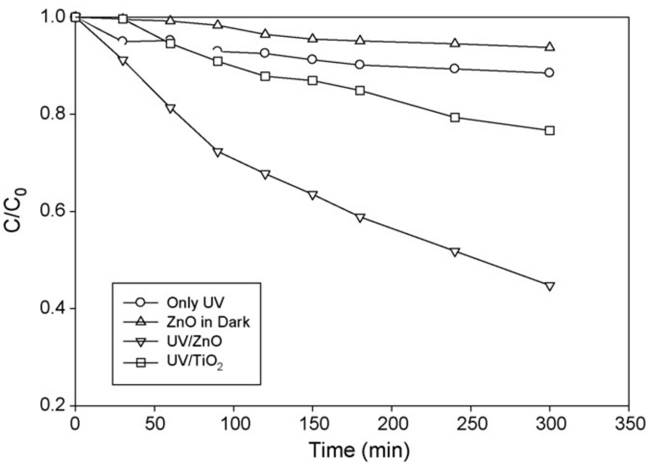

Fig. 1. Preliminary test of phenol oxidation under UV and ZnO. Reaction conditions: [phenol] = 25 ppm, $T = 25\ ^{\circ}C$, [TiO_2] = [ZnO] = 0.2 g/L, UV intensity = 2.8 W/m^2.

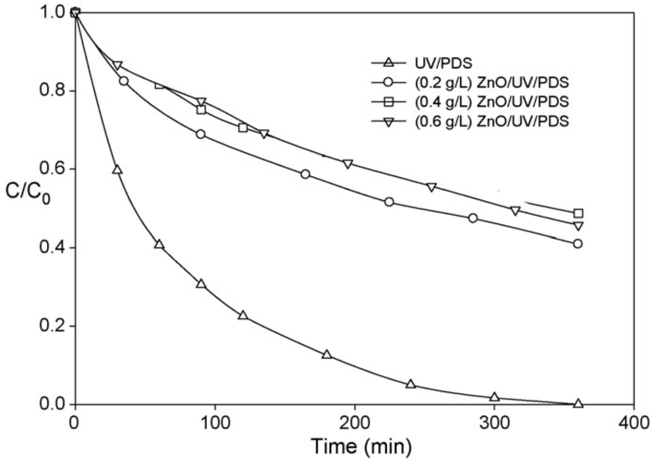

Fig. 2. Phenol oxidation with combination of low-power UV/ZnO/PDS with different amounts of ZnO. Reaction conditions: [phenol] = 25 ppm, $T = 25\ ^{\circ}C$, [PDS] = 7.4×10^{-3} M, UV intensity = 2.8 W/m^2.

Fig. 2 shows the dynamic variation of phenol concentration under photochemical reaction (UV/PDS) and the effect of addition of ZnO in solution. Under UV/PDS, phenol degradation rate was high and complete phenol removal was obtained within 5 h. However, on addition of 0.2 g/L of ZnO into the reaction mixture, the rate of phenol degradation was found to decrease, showing just 60% degradation within the time span of 5 h. Furthermore, increasing the amount of the photocatalyst suppressed the rate of phenol degradation, indicating a non-synergetic effect from the combination of photochemical and photocatalytic oxidations. Interestingly, it was observed that degradation rate of phenol in the presence of 0.2 g/L of ZnO (Fig. 2) followed similarly to the degradation of phenol in the presence of 0.2 g/L of ZnO as shown in Fig. 1, thus suggesting that upon addition of ZnO to the photochemical reaction system, the reaction proceeded mainly due to the photocatalytic reaction and the effect of PDS was completely suppressed.

Under UV radiation (200–310 nm), PDS disintegrates to generate active sulphate radicals (Eq. (1)), which have an oxidation potential of 2.4 eV, thus capable of oxidising the contaminants in water.

$$S_2O_8{}^{2-} + h\gamma \rightarrow SO_4{}^{\bullet} + SO_4{}^{-} \tag{1}$$

The results from Figs. 1–2 suggest that under the given conditions, homogeneous oxidation of phenol is much more efficient than the heterogeneous oxidation with ZnO. In the combination system (UV/ZnO/PDS), the rate of oxidation is similar to that of photocatalytic oxidation using UV/ZnO. The decreased rate of phenol degradation in the combined UV/PDS/ZnO system compared to UV/PDS suggests that the photochemical oxidation of phenol occurring by the direct absorption of UV radiation by the oxidant is completely suppressed upon adding the photocatalyst. A contradicting observation, however, has been previously reported in the literature. Dhanalakshmi et al. [23] investigated phenol degradation by coupling PDS with TiO_2 and obtained a significant enhancement in the rate of phenol oxidation using a UV lamp source of 250 W. Barakat et al. [24] studied the photocatalytic oxidation of phenol and monochlorophenol in aqueous suspensions of a commercial TiO_2 rutile and found enhanced photocatalytic oxidation rate in $UV/TiO_2/H_2O_2$ system compared to either UV/TiO_2 or UV/H_2O_2 alone under a UV irradiation of 260 W/m^2. A similar observation was also reported for the oxidation of an organophosphorous pesticide with TiO_2 and H_2O_2 at a UV power of 100–450 W [25].

Interestingly in all the reported cases a high-powered UV lamp source was utilised for carrying out the experiments. In a distinctive study, Wang et al. [26] reported a non-synergetic combination of H_2O_2 with TiO_2 for 2-chlorobiphenyl degradation under the UV irradiation of 19 W/m^2. It has been claimed that the oxidant sorption on TiO_2 surface could result in significant scavenging of active holes along with quenching of OH radicals formed on the TiO_2 surface to decrease the reaction rate. A similar effect cannot be ruled out in the current case of the UV/ZnO/PDS system. However, our previous study using a high-power UV light (500 W) observed an enhanced rate of phenol degradation in the combination of photochemical and photocatalytic oxidation [22], thereby implying that the scavenging of the oxidative valance band holes by the oxidant is not the controlling factor for the decrease in oxidation rate. The reduction in the degradation rate under the UV/ZnO/PDS system is the result of the opacity caused by addition of ZnO, which could block the low-power UV radiation (intensity ∼2.8 W/m^2) penetrating the reaction mixture to induce photochemical reaction. Thus, it is deduced that a synergistic effect in UV/ZnO/PDS would be observed only beyond a certain threshold of light intensity, at which the light is capable of penetrating the opacity of dispersed ZnO particles. Additionally, the increased amount of ZnO in the UV/PDS/ZnO system, showing a reduced degradation rate even in a comparison to heterogeneous UV/ZnO, implies the opacity effect can prevail over the photochemical and photocatalytic oxidation of phenol. Therefore, the synergy effect of photochemical and photocatalytic oxidation is dependent on ZnO loading in the solution and UV radiation intensity. The synergy effect will be observed only when absorption of photons by ZnO catalyst is high enough to induce photochemical reaction.

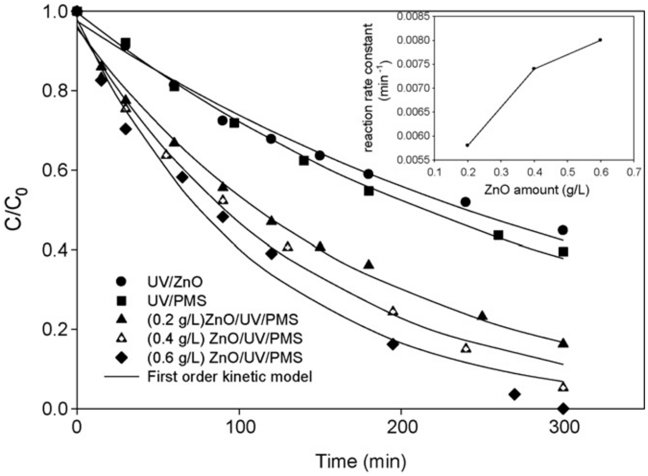

Fig. 3. Phenol oxidation with combination of low-power UV/ZnO/PMS with different amounts of ZnO. Reaction conditions: [phenol] = 25 ppm, $T = 25\,^{\circ}C$, [PMS] = 6.51×10^{-3} M, UV intensity = 2.8 W/m^2.

3.2. *Photocatalytic degradation of phenol with PMS*

Similar to PDS, PMS is known to undergo degradation upon UV irradiation (<260 nm) to generate sulphate and hydroxyl radicals thereby aiding in the photochemical oxidation of phenol (Eq. (2)).

$$HSO_5{}^{-} + h\gamma \rightarrow OH^{\bullet} + SO_4{}^{\bullet -} \tag{2}$$

Fig. 3 depicts the kinetic degradation of phenol under various reaction conditions. With UV/PMS, phenol degradation was almost 60% in 5 h, which is higher than the photocatalytic UV/ZnO oxidation. The combination of UV/PMS with ZnO resulted in an enhancement of the degradation rate to almost 80% phenol degradation within 5 h, thereby suggesting a synergistic effect with UV/ZnO/PMS, contrary to the previous UV/ZnO/PDS results. Furthermore, an increase in ZnO loading in the solution increased the degradation rate and phenol degradation would be 100% at 0.6 g/L of ZnO.

The enhanced phenol degradation in UV/PMS/ZnO system compared to UV/PMS system suggests that the contribution of photochemical oxidation was still significant in phenol degradation unlike the combination of PDS with ZnO. For UV/ZnO/PMS, it was observed that some ZnO particles dissolved in the solution during the reaction process, which made the reaction mixture more transparent and allowed sufficient transmission of UV light to induce photochemical oxidation with PMS.

Apart from photochemical and photocatalytic reaction taking place in UV/PMS/ZnO system, a series of additional reactions were found to occur due to the direct reaction of ZnO with PMS, which may also cause the dissolution of ZnO. In order to investigate the effect of ZnO dissolution and its possible influence on phenol degradation, several experiments were performed with ZnO and PMS in the absence of UV radiation. As seen in Fig. 4, phenol degradation occurred in the presence of ZnO with PMS without UV radiation. Furthermore the rate of degradation increased with the increase in concentration of ZnO. However, the control experiment conducted in the absence of ZnO showed negligible phenol degradation. For ZnO/PDS, no such reaction was observed. Previous investigations have found that PMS can be activated by transition metals to produce sulphate radicals which can then degrade organic compounds in water. Similarly, in the current case, PMS was activated by ZnO to generate active hydroxyl and sulphate radicals (Eq. (3)).

$$ZnO + HSO_5{}^{-} \rightarrow Zn^{2+} + OH^{\bullet} + SO_5{}^{\bullet -} \tag{3}$$

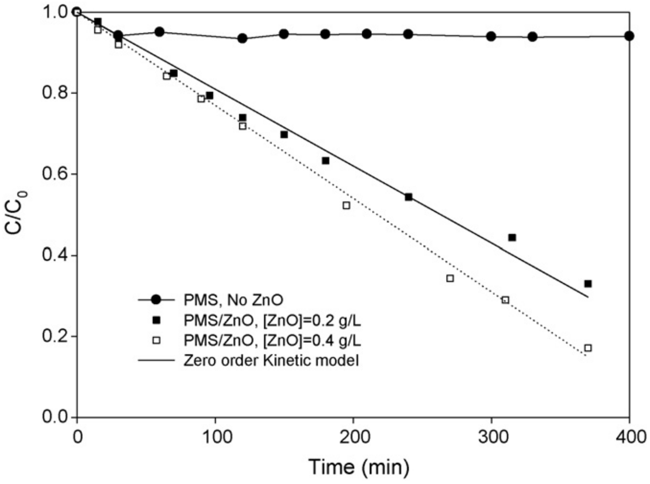

Fig. 4. Phenol oxidation with ZnO/PMS under dark conditions. Reaction conditions: [phenol] = 25 ppm, $T = 25\,^\circ C$, [PMS] = 6.51×10^{-3} M, UV intensity = $2.8\ W/m^2$.

Table 1
Rate constant of phenol degradation in PMS and ZnO.

Amount of ZnO (g)	Rate constant (mg/L min)
0.2	0.0018
0.4	0.0023

The rate of phenol degradation was found to follow zero-order kinetics, thus implying that the phenol oxidation rate is controlled by the generation of active sulphate/hydroxyl radicals rather than by the reaction with phenol itself. The rate constant of phenol degradation based on the zero-order is given in Table 1. It is shown that the rate of phenol photodegradation is enhanced at increased ZnO loading. However such an enhancement of the reaction results in the loss of the photocatalytst.

Given the series of reactions taking place in UV/PMS/ZnO, the photocatalytic oxidation of phenol in UV/PMS/ZnO system would generally occur following these mechanisms:

(1) homogeneous photochemical oxidation by UV/PMS and sulphate radicals generated from the interaction of PMS with conduction band electrons,
(2) heterogeneous photocatalytic oxidation from UV/ZnO, and
(3) chemical reaction between ZnO and PMS to generate active sulphate or hydroxyl radicals.

Fig. 5 shows the effect of PMS concentration on photodegradation of phenol in UV/ZnO/PMS. The incremental addition of PMS from 1.85 to 7.4 mM resulted in increased phenol degradation by reaching a maximum of 100% within 6 h. Further addition of PMS, however, reduced the extent of phenol oxidation efficiency. This can be attributed to two factors. One is the dissolution of ZnO with PMS causing the reduction in oxidation rate. The other factor is the self-quenching of sulphate and hydroxyl radical by PMS (Eqs. (4) and (5)). It is known that $SO_5^{\bullet -}$ exhibits lower oxidation rate than $SO_4^{\bullet -}$. Previous investigations also have found that higher PMS in metal/PMS systems would reduce organic degradation in water [27]. Therefore, an optimum amount of PMS/ZnO is important and required for the synergistic behaviour in the UV/ZnO/PMS photocatalytic oxidation of contaminants.

$$HSO_5^- + SO_4^{\bullet -} \rightarrow SO_5^{\bullet -} + SO_4^{2-} + H^+ \qquad (4)$$

$$HSO_5^- + OH^\bullet \rightarrow SO_5^{\bullet -} + H_2O \qquad (5)$$

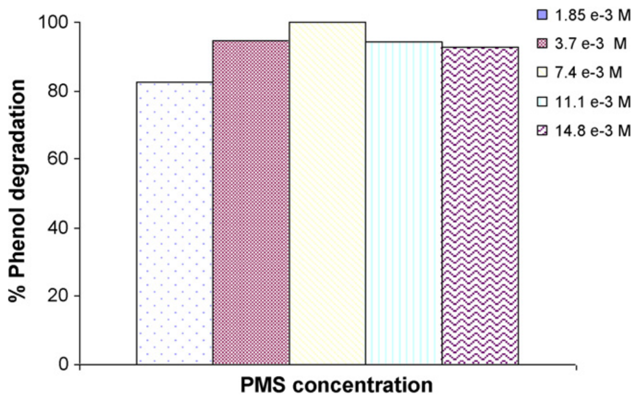

Fig. 5. Phenol oxidation efficiency under UV/ZnO/PMS for a fixed reaction time of 5 h and different concentrations of PMS. Reaction conditions: [phenol] = 25 ppm, $T = 25\,^\circ C$, [ZnO] = 0.4 g/L, UV intensity = $2.8\ W/m^2$.

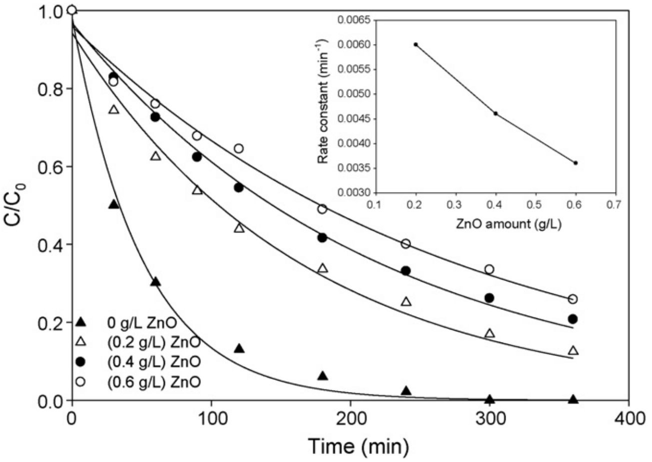

Fig. 6. Phenol oxidation with combination of low-power UV/ZnO/H_2O_2 with different amounts of ZnO. Reaction conditions: [phenol] = 25 ppm, $T = 25\,^\circ C$, [H_2O_2] = 6.68×10^{-3} M, UV intensity = $2.8\ W/m^2$.

3.3. *Photocatalytic degradation of phenol with H_2O_2*

Fig. 6 shows the dynamic variation of phenol degradation in H_2O_2, UV/H_2O_2 and UV/ZnO/H_2O_2 systems. H_2O_2 in dark conditions did not produce significant phenol degradation. With the assistance of UV, phenol degradation was much stronger and the phenol degradation reached 100% in 5 h. However, the rate of phenol degradation with UV/ZnO/H_2O_2 was reduced in a comparison to UV/H_2O_2. Lower phenol degradation rates were observed at increased ZnO loading in solution. The degradation rate reduced linearly with the increase in the amount of ZnO. The reduced phenol degradation could still be attributed to ZnO dispersion in the solution making the solution turning milky and preventing UV radiation to penetrate. Therefore, for UV/ZnO/H_2O_2, it is also suggested that the synergistic combination of UV/ZnO with UV/H_2O_2 would be realised by employing a lamp with the intensity above a minimum threshold.

With regard to the effect of H_2O_2 concentration on photocatalytic oxidation of phenol in UV/ZnO/H_2O_2 (Fig. 7), the increase in H_2O_2 concentration resulted in a substantial increase in phenol oxidation rates to a maximum limit. Further increases in H_2O_2 concentration actually reduced phenol oxidation efficiency. This is due to quenching of the active hydroxyl radicals by H_2O_2 (Eq. (6)), similar to the quench of sulphate radicals. Many investigators have

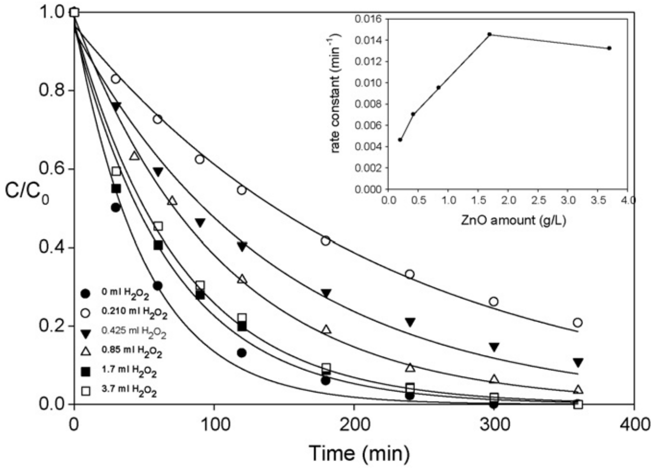

Fig. 7. Phenol oxidation with combination of low-power UV/ZnO/H_2O_2 at different concentrations of H_2O_2. Reaction conditions: [phenol] = 25 ppm, T = 25 °C, [ZnO] = 0.4 g/L, UV intensity = 2.8 W/m^2.

reported the quench effect of excess H_2O_2 [26,28].

$$H_2O_2 + OH^\bullet \rightarrow HO_2{}^\bullet + H_2O \qquad (6)$$

4. Conclusions

ZnO is a promising photocatalyst as an alternative to TiO_2 and shows effective oxidation of phenol under low-powered UV radiation. Three oxidants, PMS, PDS and H_2O_2, are also effective in photochemical degradation of phenolic pollutants and exhibited higher activity than the heterogeneous photocatalytic oxidation using UV/ZnO, under a given lamp source. However, the combination of photochemical and photocatalytic oxidation can produce different synergistic effects depending on the oxidant, ZnO loading, and UV power strength. Under low-power UV radiation, reduced phenol degradation was observed for UV/PDS/ZnO and UV/H_2O_2/ZnO systems when compared to their respective photochemical reactions, due to the blockage and scattering of UV radiation by ZnO particles. This implies that the synergistic effect could be observed only after a certain threshold of light intensity was reached to have enough photon absorption. A similar observation was found for the combination of photocatalytic UV/ZnO reaction with UV/H_2O_2 photochemical reaction. In the case of PMS-based system, the combination of UV/ZnO and UV/PMS showed a higher reaction rate compared to UV/PMS, even at low UV power. This was due to the direct reaction between ZnO and PMS, resulting in partial ZnO dissolution and better UV transmission as well as the generation of active radicals. Kinetic studies showed that photocatalytic oxidation of phenol followed the first-order kinetics while the catalytic oxidation of phenol in ZnO/PMS followed zero-order kinetics.

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