

## **Oxidation of Catechol and Hydroquinone in Aqueous Media by Heterogeneous Photocatalysis Using Thin Layer of TiO<sub>2</sub> P25**

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### **Authors' contributions**

*This work was carried out in collaboration between all authors. Authors GDB and LMB designed the study. Author DH performed the research and managed the analyses of the study. The research in lab was done under the supervision of authors TK, AAD and IT. Authors DH, AAD and TK performed the statistical analysis, wrote the protocol, managed the literature searches and wrote the first draft of the manuscript. All authors read and approved the final manuscript.*

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### **ABSTRACT**

Catechol and hydroquinone, two phenolic compounds, have been studied. The oxidation of the two chemicals by heterogeneous photocatalysis TiO<sub>2</sub> P25 Degussa based was investigated using a laboratory scale reactor and ultraviolet-A lamp. Thin layers of TiO<sub>2</sub> on stainless steel were prepared by electrophoretic deposition. Phenolic compounds were monitored by High- Performance Liquid Chromatography (HPLC) system equipped with a phenyl Xbridge column and Photodiode Array (PDA) detector. The effects of some parameters such as initial concentration of phenolic compounds (50–150 μM), initial pH (2–11) and the degradation kinetics of the two chemicals have been evaluated. The results showed that the degradation rate of catechol and hydroquinone

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decrease when the initial concentration of pollutants increases whereas the natural pH (5.7 and 4.8 respectively for catechol and hydroquinone) has been found favourable for the degradation of the two chemicals. The first order kinetic fits the oxidation rate of the two chemicals using Langmuir–Hinshelwood model. Two intermediate by-products have been identified: by-product absorbing at the wavelength of 246 nm (1,4-benzoquinone) identified in the case of hydroquinone photodegradation and another intermediate by-product absorbing at the wavelength of 256 nm (probably hydroxybenzoquinone).

*Keywords: Phenolic compounds; photooxidation; stainless steel; thin layers.*

## 1. INTRODUCTION

Phenol and its derivatives (catechol, hydroquinone, resorcinol etc.) are widely used in the industries. For example, catechol (1,2-dihydroxybenzene, CC) and hydroquinone (1,4-dihydroxybenzene, HQ) are two significant isomers of dihydroxybenzene, which have been widely used in the production of dyes, photo stabiliser, plasticisers, cosmetics, pesticides and some pharmaceuticals [1]. Thus, phenolic compounds are generally considered to be one of the important organic pollutants discharged into the environment causing unpleasant taste and odour in drinking water because of their high solubility in water [2]. Biological degradation, chlorination or ozonation, are not efficient to remove these compounds from water due to their toxicity closely linked to aromatic ring [3].

Advanced oxidation processes (AOPs) can be an alternative for the effective removal of these pollutants from liquid effluents since these methods lead to the mineralisation of organic compounds. Among the AOPs, heterogeneous photocatalytic oxidation using  $\text{TiO}_2$  as a catalyst is widely studied. Titanium dioxide is the most popular photocatalyst with good activity under UV light [4], used in numerous fields [5], proved to be an ideal photocatalyst due to its ability to completely mineralise the target pollutants [6] and this material is abundant [7]. However, some inconvenience subsist such as  $\text{TiO}_2$  spectrum absorption is limited in UV [8], and slurry form causes sedimentation and enhances recombination of photoinduced electron-hole pairs [9]. To avoid the last problem,  $\text{TiO}_2$  can be deposited as a thin film onto appropriated support such as stainless steel or glass.

The study aims to test the efficiency of  $\text{TiO}_2$  P25, as a thin film, on catechol and hydroquinone removal by heterogeneous photocatalysis process and to investigate the influence of some parameters such as the pH, the initial pollutant

concentration, and the kinetics of the phenolic compounds photooxidation.

## 2. MATERIALS AND METHODS

All chemical products were used without purification. Catechol and hydroquinone have been obtained from Sigma Aldrich. Hydrogen peroxide (30% w/w), acetylacetone (99.5%) and surfactant Triton X 100 are from Fischer Scientific.  $\text{TiO}_2$ P25 with 80% anatase and 20% rutile was obtained from Evonik. The sol- gel suspension of  $\text{TiO}_2$  was prepared according to the method of Kodom et al. [10]. Thin layers of  $\text{TiO}_2$  were prepared by electrophoretic deposition from a sol- gel suspension. Stainless steel used as support (cathode) was cleaned carefully with a mixture of hydrogen peroxide and sulphuric acid and washed several times with distilled water. A voltage of 10 V has been applied to anode and cathode plates, separated by a length of 1 cm to ensure an electrical field causing the movement of  $\text{TiO}_2$  particles toward the cathodic plate. This voltage is applied during 40 s. The obtained deposits are then dried in air and annealed in an oven at 450 °C with 5°C/min as speed for 1 h heating to avoid structural modification of catalyst. The removal of Catechol and hydroquinone by photo oxidation was carried out in a reactor as reported by Dougna et al. [11].

Comparative studies of photolysis, adsorption and photocatalysis as well as some parameters (concentration and pH) have been performed. Before turning on the lamp for photocatalysis studies, adsorption study was done. Kinetic study of catechol and hydroquinone degradation has been done using Langmuir- Hinshelwood model since degradation has been performed with low concentration.

HPLC system (Alliance Waters 2690, Waters) coupled to a diode array detector (Waters 2996, PDA) with an Xbridge column of phenyl type (150 mm x 4.6 mm, 5 mm) was used for the

identification and quantification of the phenolic compounds in aqueous solution [11]. The abatement rate (A), has been calculated using the following equation.

$$A = (C_0 - C) \times 100 / C_0 \quad (1)$$

where  $C_0$  is the initial concentration of pollutant and C the residual concentration at time t.

### 3. RESULTS AND DISCUSSION

#### 3.1 Catechol and Hydroquinone Degradation

##### 3.1.1 Comparative study of adsorption, photolysis and photocatalysis

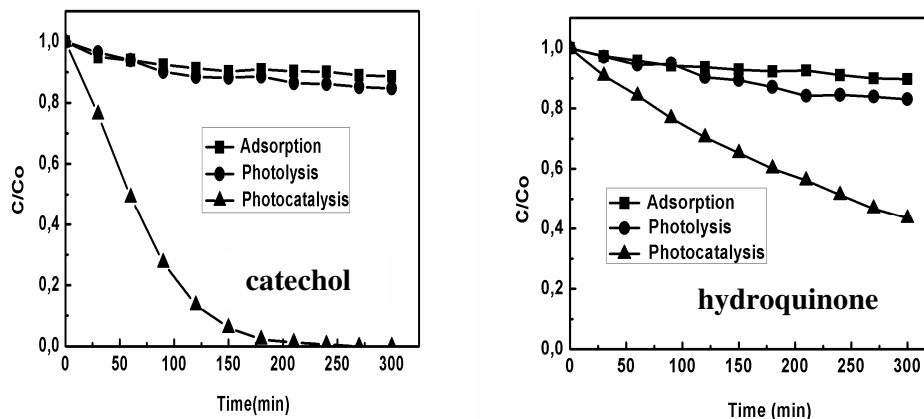
The adsorption (catalyst without UV light), the photolysis (UV light without catalyst) and the photocatalysis (UV light and catalyst) have been performed on each phenolic compound in aqueous solution at natural pH and with a concentration of 50  $\mu\text{M}$  to assess the efficiency of the three processes (Fig. 1). The removal rate of 11.4%, 15.3% and 100% have been obtained respectively for the adsorption, the photolysis and the photocatalysis for catechol, whereas 10.3%, 16.9% and 57% have been received for the three processes for hydroquinone after 300 min (Table 1).

These results show that the processes of photolysis and adsorption contribute slightly to the removal of catechol and hydroquinone from aqueous solution (less than 20%) compared to the rate of the photocatalysis during the same time. The same observation has been reported by Dougna et al. [11] and Hermann J [12] for phenol removal by photocatalysis using  $\text{TiO}_2$ . The oxidation by the photocatalysis is more efficient than the adsorption and the photolysis in the experimental condition. Nevertheless, the rate of the photolysis is higher than those obtained in the literature [13]. Taking into account, the contribution of photolysis during photooxidation process, the removal rate of pollutants by photocatalysis was 84.7% and 40.1% respectively for catechol and hydroquinone. Thus, the relatively slight contribution of photolysis and adsorption to catechol and hydroquinone removals revealed a significant photocatalytic activity.

The abatement rate obtained is remarkably higher than those obtained by Dougna et al. [11] on the phenol photocatalysis. The reason for this can be correlated to the nature of the substrate and also the influence of the initial concentration. Indeed, they had used a concentration of 100  $\mu\text{M}$  whereas the present study used 50  $\mu\text{M}$ .

**Table 1. Abatement rate of the three processes for catechol and hydroquinone removal**

	Catechol		Hydroquinone	
Time (min)	180	300	180	300
Adsorption (50 $\mu\text{M}$ )	9,1%	11,4%	7,7%	10,3%
Photolysis (50 $\mu\text{M}$ )	11,4%	15,3%	12,9%	16,9%
Photocatalysis (50 $\mu\text{M}$ )	97,8%	100%	40%	57%
Photocatalysis without photolysis	86,4%	84,7%	27,1%	40,1%



**Fig. 1. Adsorption, photolysis and photocatalysis at natural pH and concentration of pollutants 50  $\mu\text{M}$**

### 3.1.2 Effect of initial concentration

In this study, three different concentrations (50  $\mu\text{M}$ , 100  $\mu\text{M}$  and 150  $\mu\text{M}$ ) have been used to evaluate the effect of initial concentration of the pollutants on the photodegradation process. The result (Fig. 2) reveals that the degradation is more efficient for the lowest concentrations (50  $\mu\text{M}$  > 100  $\mu\text{M}$  > 150  $\mu\text{M}$ ). This observation was confirmed by the rate of abatement of the pollutants calculated at a different time for the two pollutants (Table 2). The abatement rate decreased when the initial concentration increased, however, the removed amount increased with the concentration. These observations have been supported by previous authors such as Saquib and Muneer [14] on triphenylmethane dye photocatalytic degradation, Konstantinou et al. [15] on azo dyes photocatalytic degradation and Dougna et al. [11] on phenol removal by photocatalysis. This result can be explained by the fact that as the initial concentration of the pollutants increases, more and more pollutant molecules are adsorbed on the surface of the catalyst. Hence, the penetration of light to the surface of the catalyst decreases leading to lowering in the production of oxidation species such as  $\text{OH}^\circ$  and  $\text{O}^{2-}$  [14]. An increase of initial pollutants concentration also decreases the path length of photons entering into the pollutants solution. At high pollutants concentration, molecules may absorb a little amount of UV radiation rather than the catalyst, and this may also reduce the catalytic efficiency [3]. A large amount of pollutant might cause poisoning of catalyst surface. For catechol, abatement decreases significantly between 100  $\mu\text{M}$  and 150  $\mu\text{M}$  at 180 min but further increase at 300 min and reaches the similar amount removed for 100  $\mu\text{M}$  and 150  $\mu\text{M}$  (Table 2). In the case of hydroquinone, changes are different showing that the mechanisms of removal are not the same. Indeed, pollutant nature and reaction time affect significantly photocatalytic activity.

### 3.1.3 Effect of pH

The pH value is one of the parameters that influence the rate of degradation of some organic compounds in the photocatalytic process. The effect of pH on phenolic compounds photocatalysis was investigated in this study in the range of 2 and 11 with a fixed concentration of 50  $\mu\text{M}$ . The result (Fig. 3) reveals that the natural pH (5.7 and 4.8 for catechol and hydroquinone respectively) was found to be

favourable for the phenolic compounds degradation with the highest abatement rate of 100% and 57% respectively for catechol and hydroquinone after 300 min. The pH 11 was less favourable for phenolic compounds removal as shown in Fig. 3. A similar observation has been reported by Pelentridou et al. [16] on the effect of pH on the photodegradation of the herbicide azimsulfuron and also by Dougna et al. [11] on phenol removal by photocatalysis. This result can be explained by the repulsion forces between the catalyst surface charge and the charge of pollutants molecules. Indeed, the  $\text{pH}_{\text{pzc}}$  of  $\text{TiO}_2$  is about 6.8 [17], and the  $\text{pK}_a$  of the phenolic compounds is about 9.48 and 10.85 respectively for catechol and hydroquinone. The presumably positively charged acidic media ( $\text{pH} < 6.8$ ) and negatively charged basic media ( $\text{pH} > 6.8$ ) will promote or inhibit the establishment of Coulomb electrostatic strength during adsorption and can thus influence the interaction between the catalyst and the pollutant surfaces [11]. The pH plays a significant role in photocatalysis based on  $\text{TiO}_2$ , so its effect on the photocatalytic efficiency must be accurately checked before any application. Working at free pH can be very interesting since the process will not need acid or alkali for pH modifications which can increase operating cost.

### 3.2 Kinetic Study of Catechol and Hydroquinone Removal by Photooxidation

A fixed concentration of 50  $\mu\text{M}$  and free pH of the solution have been used for degradation kinetic study. Langmuir- Hinshelwood model has been used to describe the degradation kinetic since the concentration used was less than  $10^{-3}$  M [18]. The first apparent order constant  $k_{\text{app}}$  can be calculated according to the following equation [3]:

$$-dC/dt = k_{\text{app}}C \quad (2)$$

$\ln(C_0/C)$  versus time will give a straight line, and the slope of this line upon linear regression corresponds to the apparent first-order rate constant  $k_{\text{app}}$  (Fig. 4).

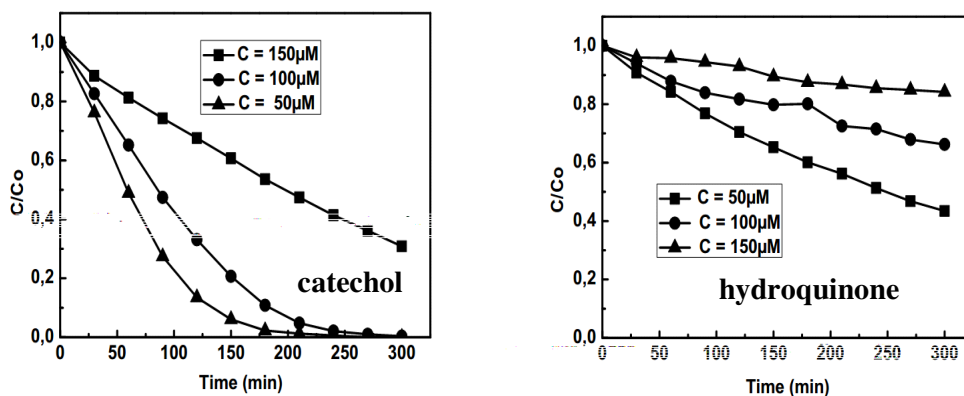
The analysis of the data given in Table 3 and Fig. 5 shows that the apparent first order constant  $k_{\text{app}}$  decreases when the pollutants concentration increases and for the same concentration this constant increases with the time. The same observation has been reported by other authors such as Mozia et al. [3] and Galindo et al. [19]. For example, Chiou et al. [20] reported that the

first-order rate constant  $k_{app}$  during phenol photodegradation decreases with initial phenol concentrations. The presumed reason is that at high pollutants concentrations the generation of  $^{\circ}OH$  radicals on the surface of a catalyst is reduced since the active sites are covered by pollutants. However, over the time, pollutant concentrations are significantly reduced leading to an increase in removal rate, but it highly depends on the pollutant nature and reaction time (Table 2).

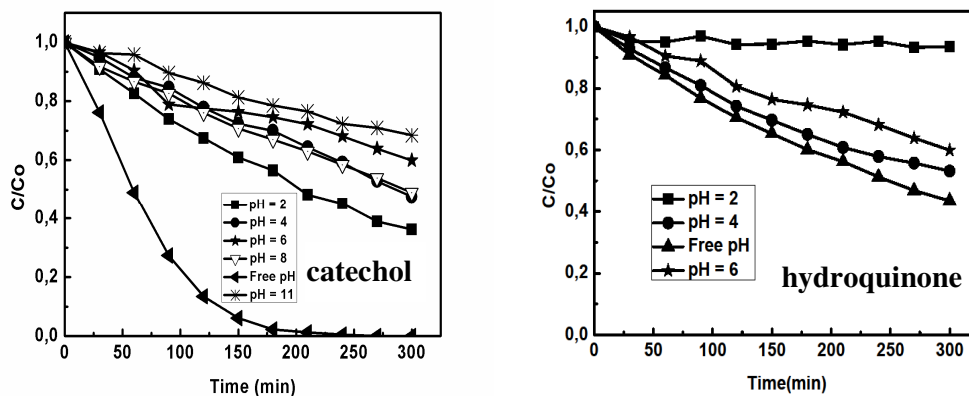
The relative weak value of the  $k_{app}$  obtained in the case of hydroquinone can be linked to a difference in behaviour of the two chemicals toward the photodegradation. The catechol is more sensible than hydroquinone for photocatalytic removal using  $TiO_2$  P25. Indeed, according to Holleman rules [21], hydroquinone is more stable than catechol due to steric hindrance occurring in catechol and making easier its oxidation. More a chemical is stable, more difficult is its degradation.

**Table 2. Abatement rate (A, %) and removal concentration (RC,  $\mu M$ ) at different time**

	Catechol				Hydroquinone			
	A (%)	RC ( $\mu M$ )	A (%)	RC ( $\mu M$ )	A (%)	RC ( $\mu M$ )	A (%)	RC ( $\mu M$ )
Time (min)	180	300	-	180	-	300	-	-
Photocatalysis (50 $\mu M$ )	97.8	48.9	100	50	40.0	20	57.0	28,5
Photocatalysis (100 $\mu M$ )	89.3	89.3	99.7	99.7	19.9	19.9	33.8	33.8
Photocatalysis (150 $\mu M$ )	39.2	58.8	66.4	99.6	12.5	18.8	15.9	23.85



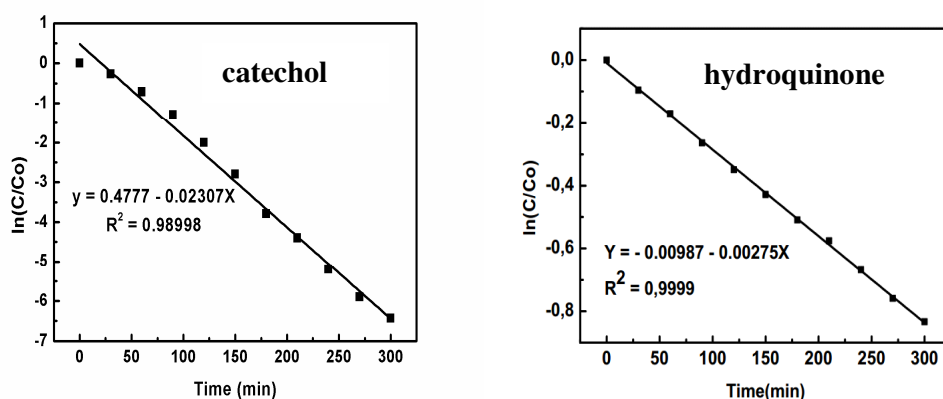
**Fig. 2. Effect of the initial concentration on the pollutants removal at natural pH (5.7 and 4.8 for catechol and hydroquinone respectively)**



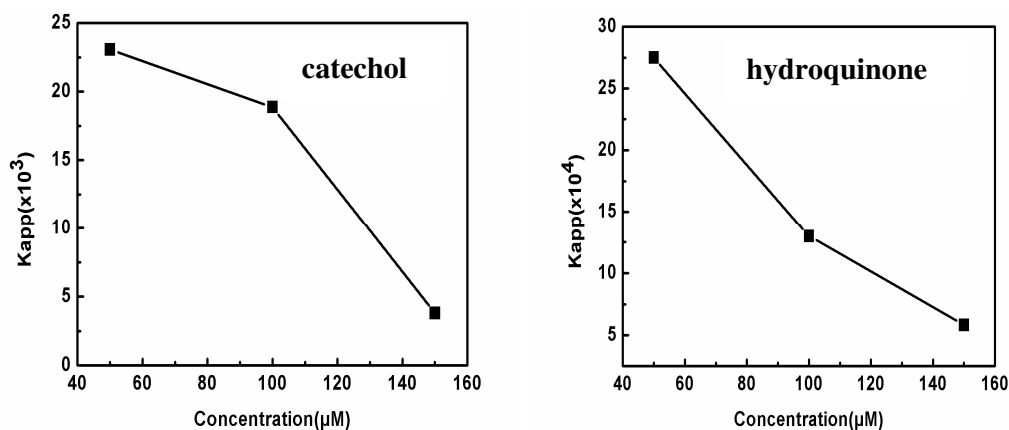
**Fig. 3. Effect of pH on catechol photocatalysis (C = 50  $\mu M$ ) at natural pH (5.7 and 4.8 for catechol and hydroquinone respectively)**

**Table 3. First order apparent constant kinetic  $k_{app}$  for different concentration at different time**

	First order apparent constant kinetic $k_{app}$ ( $\times 10^3$ ) ( $\text{min}^{-1}$ ); $R^2$			
	Catechol		Hydroquinone	
Time (min)	180	300	180	300
Photocatalysis (50 $\mu\text{M}$ )	21.07; 0.98	21.44; 0.98	2.77; 0.99	2.91 ; 0.99
Photocatalysis (100 $\mu\text{M}$ )	12.37; 0.94	18.99; 0.94	1.37 ; 0.96	1.68 ; 0.96
Photocatalysis (150 $\mu\text{M}$ )	3.46; 0.99	3.91; 0.99	0.574 ; 0.964	0.615 ; 0.964



**Fig. 4. Apparent first order linear transform  $\ln(C/C_0) = f(t)$  for photocatalytic decomposition of phenolic compounds (50  $\mu\text{M}$ ); reaction temperature: 303 K**



**Fig. 5. Apparent constant kinetic  $k_{app}$  evolution against pollutants concentration**

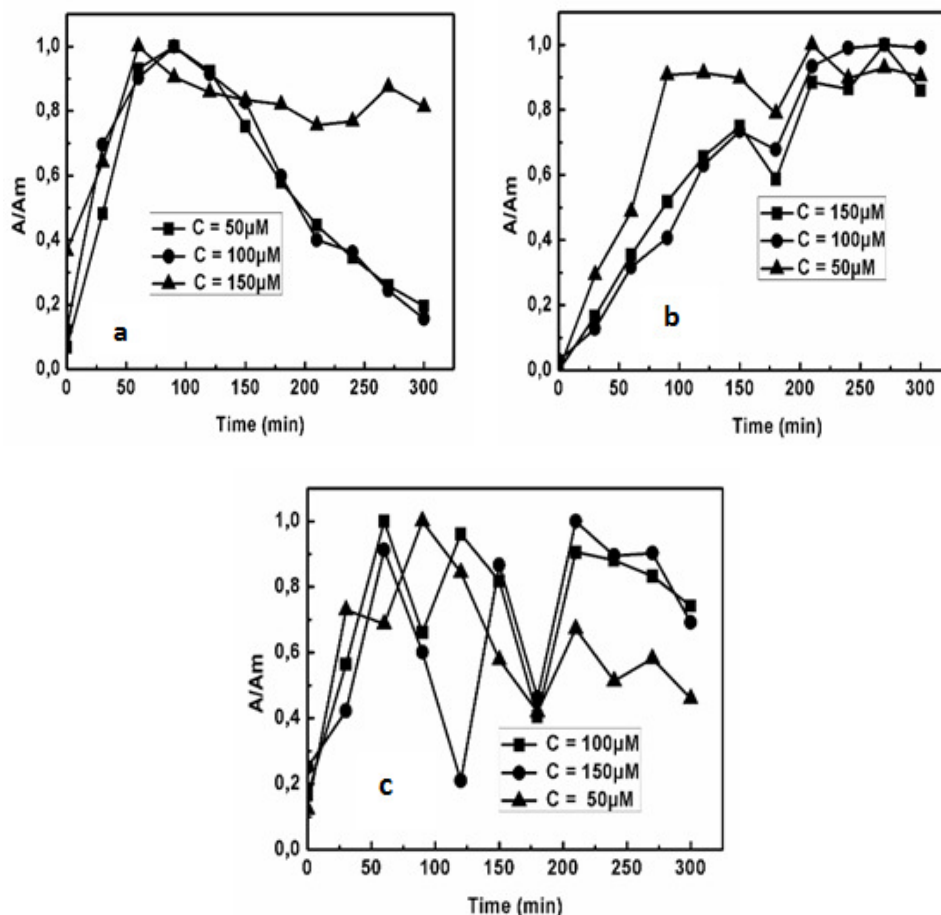
### 3.3 Intermediate By-products of Catechol and Hydroquinone during Their Photodegradation

The key advantage of Advanced Oxidation Processes is its ability to degrade organics towards mineralisation [15]. However, mineralisation or transient intermediate production is usually none investigated. In this study, concerning catechol and hydroquinone removal, two by- products have been noted

during the monitoring of the remained concentration of the phenolic compounds in aqueous media by HPLC using retention time and absorbance spectrum. One by-product appeared at the wavelength of 246 nm with a retention time of 6.5 min only during the oxidation of hydroquinone and another one at the wavelength of 256 nm and with a retention time of 5.6 min appeared during the photodegradation of the two compounds. Fig. 6 shows time course for intermediates appearing during photo

oxidation of catechol and hydroquinone where  $A_m$  is the maximum absorbance obtained for intermediates. The first intermediate fitted well the HPLC characteristic of benzoquinone while the second by-product needed a more analytical method for identification [11]. The intermediate at 246 nm (benzoquinone) existing at the initial time, is an impurity of reversible reduction-oxidation production of hydroquinone occurring in solution [22]. Non-identified intermediate at 256 nm is also correlated with catechol initially. However, their concentration increased over the time showing that they are intermediate by-products of catechol and hydroquinone. At the industrial scale, catechol and hydroquinone are together manufactured by direct hydroxylation of phenol with peroxides followed by separation and purification methods [23]. This process could lead to residual dihydroxybenzene oxidation

products. Intermediate identified at 256 nm could not be phenolic compound due to its wavelength which is not located around 210–220 nm and 270–280 nm [24]. In their works, Fiege et al. [23] reported that by-products with a maximum wavelength of 256 nm could be 1,2-benzoquinone. However, as this by-product appears during hydroquinone oxidation, one could assume that it would be a hydroxybenzoquinone [25,26]. Indeed, hydroxybenzoquinone could be derive from 1,2,4-trihydroxybenzene, an intermediate obtained through addition of OH on catechol or hydroquinone. Fortunately, these by-products should also be subjected for photocatalytic oxidation since their chromatographic peaks decrease over the time especially in the case of catechol. This fast drop should be due to mineralisation of chemicals.



**Fig. 6. Kinetic of intermediates appearing during photodegradation of catechol and hydroquinone at natural pH, Concentrations (50, 100, 150 μmol/L)**  
*a: intermediate at 256 nm for catechol, b: intermediate at 256 nm for hydroquinone;*  
*c: intermediate at 246 nm appearing during hydroquinone photooxidation*

#### 4. CONCLUSION

The efficiency of thin layers of TiO<sub>2</sub> P25 on stainless steel for catechol and hydroquinone removal by photocatalysis has been investigated. The result shows that photodegradation efficiency of phenolic compounds was weak when photolysis and adsorption were carried out. By photo oxidation mechanism, 84.7% and 40.1% of removal rate were obtained respectively for catechol and hydroquinone (50 µM) after 300 min of the experiment. Phenolic compounds degradation was highly dependent on initial concentration and initial pH. The abatement rate and the apparent constant kinetics obtained for the two pollutants revealed that catechol and hydroquinone have different behaviour toward the photocatalysis TiO<sub>2</sub> based and the catechol is more sensible for oxidation than hydroquinone. The best condition for optimal phenolic (catechol and hydroquinone) photodegradation TiO<sub>2</sub> based imposes to operate at natural pH.

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#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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## APPENDIX



**Reactor used for catechol and hydroquinone removal by photooxidation**



**Thin layer of TiO<sub>2</sub> P25 onto stainless steel after annealing at 450 °C for 1 h**

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