

# Mechanism of phenol decomposition on Fe–C–TiO<sub>2</sub> and Fe–TiO<sub>2</sub> photocatalysts via photo-Fenton process

Beata Tryba<sup>a,b,\*</sup>, Antoni W. Morawski<sup>a</sup>, Michio Inagaki<sup>c</sup>, Masahiro Toyoda<sup>b</sup>

<sup>a</sup> Technical University of Szczecin, Chemical Technology and Environmental Engineering, Ul. Pulaskiego 10, 70-322 Szczecin, Poland

<sup>b</sup> Oita University, 700 Dannoharu, Oita 870-1192, Japan

<sup>c</sup> Aichi Institute of Technology, Yakusa, Toyota 470-0392, Japan

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## Abstract

Photoactivity of TiO<sub>2</sub> photocatalyst for phenol decomposition was enhanced through its modification by the mounting of iron. Two types of Fe–C–TiO<sub>2</sub> photocatalysts were prepared, the one having the high content of carbon, around 7.5–15.5 mass% (obtained from carbonization of poly(ethylene terephthalate) (PET)), and the other, contained the residual carbon, 0.2–3.3 mass% (obtained from the carbonization of oxalate). Photodecomposition of phenol on these Fe–C–TiO<sub>2</sub> photocatalysts proceeded through the photo-Fenton reactions and was faster on the photocatalysts with the residual carbon, which had high content of iron. The adsorption of phenol was higher on the photocatalysts having the high content of carbon. On Fe–TiO<sub>2</sub>, the phenol decomposition was accelerated insignificantly by comparison with the pristine TiO<sub>2</sub>. It was proved that different mechanisms of phenol decomposition occurred on Fe–TiO<sub>2</sub> and Fe–C–TiO<sub>2</sub>, on the former the phenol was decomposed by the radical reaction and on the latter by the complex reaction with the iron and intermediates of phenol decomposition. The radical reaction on Fe–TiO<sub>2</sub> photocatalyst is not selective for phenol decomposition, therefore phenol has been decomposed slowly. By the complex reactions of phenol oxidation on Fe–C–TiO<sub>2</sub> photocatalysts, the rate of phenol decomposition is sped up significantly, resulting in the complete mineralization of phenol in a very short time.

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**Keywords:** Phenol decomposition; Photo-Fenton reaction; Carbon-coated TiO<sub>2</sub>; FeC<sub>2</sub>O<sub>4</sub>

## 1. Introduction

Carbon-coated TiO<sub>2</sub> photocatalysts were recently developed and studied [1–6]. It was proved that carbon-coated TiO<sub>2</sub> had higher photoactivity for methylene blue decomposition in water than pristine TiO<sub>2</sub> [1–3,6]. Some advantages of carbon coating were found such as suppressing of phase transformation from anatase to rutile during heat-treatment at high temperatures, increasing the adsorption of the organic compounds on the photocatalyst surface and prevention from the interaction between TiO<sub>2</sub> particles and the organic binder during photocatalytic decomposition [1–6]. However, the phenol decomposition on carbon-coated TiO<sub>2</sub> photocatalyst could not be enhanced, therefore a chemical modification of the carbon layer was performed. It was already reported that carbon-coated TiO<sub>2</sub> photocatalyst

modified by iron could successfully decompose phenol via photo-Fenton process [7].

Photo-Fenton reactions are widely studied and used in the photocatalytic process [8–15]. The most commonly used catalyst in the photo-Fenton process is iron [8]. In the presence of Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> phenol decomposition is going very fast with and without light. When iron exists only in the form of Fe<sup>3+</sup>, Fenton reaction is limiting by the reduction of Fe<sup>3+</sup> to Fe<sup>2+</sup> in the presence of H<sub>2</sub>O<sub>2</sub>, which is less efficient than oxidation of Fe<sup>2+</sup>, but after build-up of enough amount of Fe<sup>2+</sup> the significant acceleration of degradation is obtained [12].

It was reported that the addition of hydroquinone or catechol to the reaction solution could reduce Fe<sup>3+</sup> to Fe<sup>2+</sup>, accelerating in this way decomposition of phenol by the formation of ternary HQ–Fe–H<sub>2</sub>O<sub>2</sub> [12].

It was also reported that in the degradation of phenol via photo-Fenton process, iron complex plays an important role, phenol is more likely degraded through the complex oxidation than oxidation by hydroxyl radical [13].

\* Corresponding author. Tel.: +48 91 4494277; fax: +48 91 4494686.  
E-mail address: [beata.tryba@ps.pl](mailto:beata.tryba@ps.pl) (B. Tryba).

In the present study, the comparison of phenol decomposition on Fe–C–TiO<sub>2</sub> and Fe–TiO<sub>2</sub> photocatalysts is reported. Some advantages of carbon coating on TiO<sub>2</sub> were found for mounting iron and its application as the photocatalyst in the photo-Fenton process.

## 2. Experimental

Four types of TiO<sub>2</sub> photocatalysts were prepared. Their preparation conditions are summarized in Table 1.

TiO<sub>2</sub> as-received, commercially available anatase powder ST-01 (Ishihara Co. Ltd., Japan), was used as the reference photocatalyst and also used as the pristine material for other types of modified TiO<sub>2</sub>. It was known to consist of pure anatase phase with the crystallite size of about 7 nm and the aggregates of the size of about 60 nm and to have relatively high surface area as 300 m<sup>2</sup>/g.

Fe-modified TiO<sub>2</sub>, Fe–TiO<sub>2</sub>, was prepared by heating the powder mixture of ST-01 with FeC<sub>2</sub>O<sub>4</sub> in air at 550 °C for 3 h, the selected mixing ratio was 10/1 in mass.

Two types of Fe–C–TiO<sub>2</sub> photocatalysts were prepared. Fe–C–TiO<sub>2</sub> with the residual carbon were prepared from anatase-type TiO<sub>2</sub> (ST-01, Ishihara Sangyo Co. Ltd, Japan) and FeC<sub>2</sub>O<sub>4</sub>. Suspended TiO<sub>2</sub> in FeC<sub>2</sub>O<sub>4</sub> solution (TiO<sub>2</sub>/FeC<sub>2</sub>O<sub>4</sub> ratio = 10/1 g/g) was mixed with magnetic stirrer firstly at room temperature for around 20 h, and then until the water completely evaporated at 100 °C. After drying in oven through overnight, modified TiO<sub>2</sub> thus prepared was heated at temperatures from 400 to 900 °C in Ar for 1 h. For preparation of Fe–C–TiO<sub>2</sub> photocatalysts with high carbon content, PET was added to the water solution of FeC<sub>2</sub>O<sub>4</sub> in the mass ratio of PET/FeC<sub>2</sub>O<sub>4</sub> = 10:1 and that mixture was stirred at room temperature for 20 h and then was heated up to 100 °C to evaporate the water. After drying in oven modified PET was mechanically mixed with TiO<sub>2</sub> in the mass ratio 3:7 respectively and was heat-treated at 400–800 °C in Ar for 1 h [7]. Fe–TiO<sub>2</sub> photocatalyst was prepared by mixing the powders of TiO<sub>2</sub> with FeC<sub>2</sub>O<sub>4</sub> in the mass ratio of TiO<sub>2</sub>/FeC<sub>2</sub>O<sub>4</sub> = 10:1 and heating in air at 550 °C for 3 h.

The prepared photocatalysts were characterized by BET surface area, XRD, FT-IR and the contents of iron and carbon. BET surface area was determined from the adsorption isotherm of nitrogen at 77 K. XRD analysis was performed with using Cu K $\alpha$  radiation. Content of iron in the catalysts (Fe<sup>2+</sup> and Fe<sup>3+</sup>) was determined by extraction with 6 M HCl of 5 ml for 2 h [16] and then analyzed in the UV–vis spectrophotometer by using colorimetric method. Iron detection was carried out using 2,4,6-tris(2-pyridyl)-1,3,5-triazine reagent with very high sensitivity to Fe<sup>2+</sup>. Content of carbon in Fe–C–TiO<sub>2</sub> photocatalysts was

measured by TG analysis, combustion of carbon in air by heating the sample in air atmosphere up to 1000 °C.

Photoactivity of the catalysts was measured for the phenol decomposition in water. Photocatalysts in the mass of 0.2 g/L were added to the 500 mL of phenol solution with concentration of  $2.1 \times 10^{-4}$  mol L<sup>-1</sup> in the “batch mode” reactor and that mixture was stirred for 3 h in the dark until saturation of adsorption. After adsorption, photodecomposition of phenol was performed under UV irradiation. Reactor was equipped with three black light blue fluorescent lamps of 20 W power. These lamps irradiate the light at wavelength range of 300–415 nm with maximum intensity at 352 nm. Irradiation power of UV rays inside the reactor was 2.4 mW cm<sup>-2</sup> in the wavelength range of 310–400 nm and 0.19 mW cm<sup>-2</sup> in the wavelength range of 360–470 nm. The value of pH of the reaction mixtures was around 6.1. Some experiments at pH 2.8 were also performed. Experiments were carried out under UV with or without the addition of 0.03 mol/L H<sub>2</sub>O<sub>2</sub> to the phenol solution. The changes in the phenol concentration and formation of hydroquinone (HQ), benzoquinone (BQ), catechol and short-chain compounds as the decomposition products were measured by high performance liquid chromatography (HPLC).

## 3. Results

The characteristics of TiO<sub>2</sub> and Fe–C–TiO<sub>2</sub> photocatalysts prepared with high amount of carbon are presented in Table 2.

Those samples consisted from a single anatase phase and had 7.5–15.5 mass% of carbon, higher for the lower temperature of heat-treatment. BET surface area did not differ much in those samples. The high capacity for phenol adsorption on Fe–PET400–TiO<sub>2</sub> was caused by the difference in the chemical nature of the carbon surface as reported in our previous paper [7].

The phenol decomposition performed at pH 6.1 under UV irradiation with the addition of H<sub>2</sub>O<sub>2</sub> on TiO<sub>2</sub> and Fe–C–TiO<sub>2</sub> photocatalysts with high carbon content is presented in Fig. 1.

Only sample heat-treated at 400 °C, which showed high adsorption of phenol, showed high rate of phenol decomposition under UV with H<sub>2</sub>O<sub>2</sub>, much higher than the pristine TiO<sub>2</sub> [7].

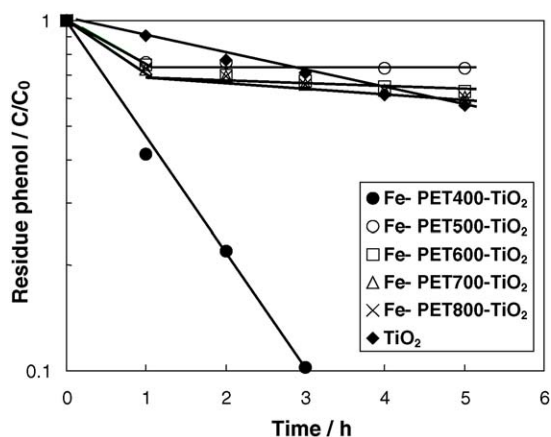


Fig. 1. Decomposition of phenol under UV irradiation and with H<sub>2</sub>O<sub>2</sub> on TiO<sub>2</sub> and Fe–C–TiO<sub>2</sub> photocatalysts with high carbon content.

Table 1  
The preparation conditions of samples

Sample	TiO <sub>2</sub> /FeC <sub>2</sub> O <sub>4</sub>	TiO <sub>2</sub> /PET	Heat treatment
TiO <sub>2</sub> (ST-01)	–	–	–
Fe–TiO <sub>2</sub>	10/1 in powder	–	550 °C in air for 3 h
Fe–C–TiO <sub>2</sub>	10/1 in water	–	400–900 °C in Ar for 1 h
Fe–PET–TiO <sub>2</sub>	10/1 in water	3/7	400–800 °C in Ar for 1 h

Table 2  
Characteristics of pristine TiO<sub>2</sub> and Fe–C–TiO<sub>2</sub> with high carbon content

HTT (°C)	Sample code	Content of TiO <sub>2</sub> (mass%)	BET surface area (m <sup>2</sup> /g)	Phases (by XRD)	Amount of Fe (mass%)		Phenol adsorption (10 <sup>-5</sup> mol/g)
					Fe <sup>2+</sup>	Fe <sup>3+</sup>	
0	TiO <sub>2</sub>	100	300	A	0	0	1.0
400	Fe–PET400–TiO <sub>2</sub>	84.5	143	A	0.33	0.01	29.2
500	Fe–PET500–TiO <sub>2</sub>	89.5	150	A	0.44	0.02	2.7
600	Fe–PET600–TiO <sub>2</sub>	90	143	A	0.43	0.07	4.2
700	Fe–PET700–TiO <sub>2</sub>	91	142	A	0.6	0.07	3.7
800	Fe–PET800–TiO <sub>2</sub>	92.5	128	A	0.46	0	5.3

Phenol decomposition performed at pH 2.8 insignificantly accelerated the rate of phenol decomposition (the results are not presented here), however the leaching of iron from the photocatalysts was noticed, what was not convenient, so all the other experiments were performed at neutral pH.

In order to investigate the role of the phenol adsorption on the surface of the Fe–C–TiO<sub>2</sub> photocatalysts in the process of phenol decomposition, some other photocatalysts were prepared with the residue carbon by mixing of FeC<sub>2</sub>O<sub>4</sub> with TiO<sub>2</sub> and heating in Ar atmosphere at different temperatures, from 400 to 900 °C, and Fe–TiO<sub>2</sub> photocatalyst was prepared by heating FeC<sub>2</sub>O<sub>4</sub> with TiO<sub>2</sub> at 550 °C in air. The characteristics of those samples are introduced in Table 3.

BET surface area was markedly reduced in those photocatalysts during the heating at high temperatures, what was caused by the sintering of TiO<sub>2</sub> particles. The content of carbon in Fe–C–TiO<sub>2</sub> photocatalysts was insignificant, ranged from 0.2 to 3.3 mass%. Transformation from anatase to rutile phase appeared at 700 °C. From the heat-treated temperature of 500 °C FeTiO<sub>3</sub> phase with ilmenite structure was clearly observed on XRD patterns of Fe–C–TiO<sub>2</sub> and Fe–TiO<sub>2</sub> photocatalysts. The highest content of iron measured by the extraction with 6 M HCl was noted for Fe–TiO<sub>2</sub> and FeC500–TiO<sub>2</sub> photocatalysts.

The adsorption of phenol on Fe–TiO<sub>2</sub> and Fe–C–TiO<sub>2</sub> with the residue carbon photocatalysts was performed before decomposition by the mixing of 0.1 g sample in 500 mL of phenol solution with concentration of  $2.1 \times 10^{-4}$  mol L<sup>-1</sup> in the dark for 3 h. These samples showed almost no adsorption of phenol.

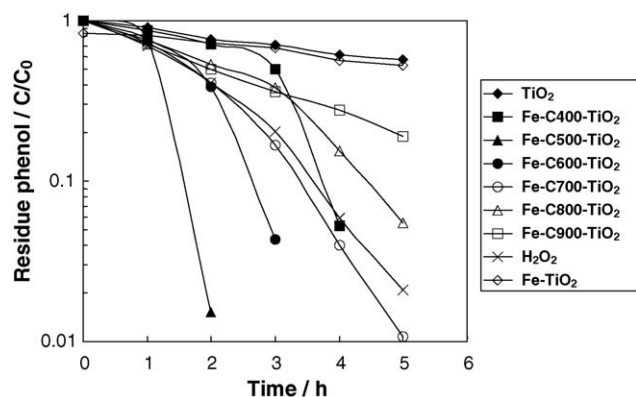
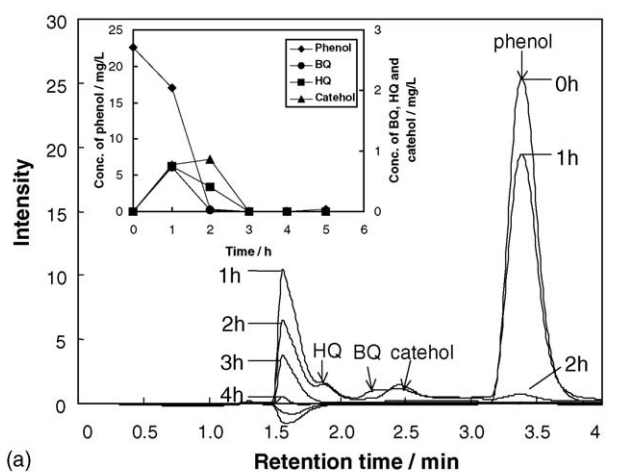
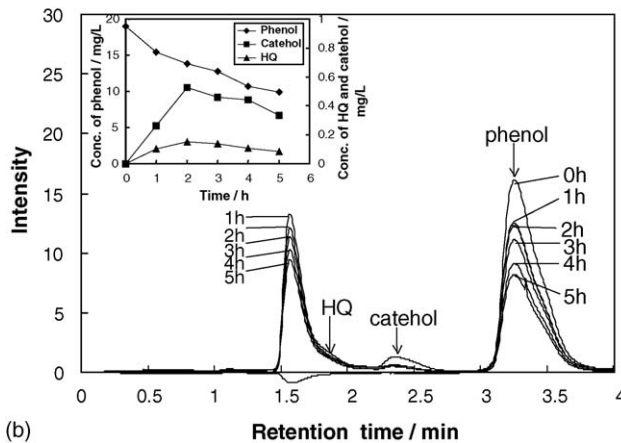


Fig. 2. Phenol decomposition under UV and with H<sub>2</sub>O<sub>2</sub> on Fe–TiO<sub>2</sub> and Fe–C–TiO<sub>2</sub> photocatalysts with residue carbon.

After adsorption, the process of phenol decomposition was performed in the same solution. In Fig. 2, the decomposition of phenol on Fe–TiO<sub>2</sub> and Fe–C–TiO<sub>2</sub> photocatalysts under UV irradiation with the addition of H<sub>2</sub>O<sub>2</sub> is presented after saturation with adsorption. The initial phenol solution had pH 6.4, after adsorption pH did not change, after addition of H<sub>2</sub>O<sub>2</sub> pH of solution dropped to 6.2 and with time of UV irradiation was insignificantly decreasing (after 1 h pH 5.3) due to the formation of decomposition products, and then was slightly increasing with following process of mineralisation (after 5 h pH 5.8).



(a)



(b)

Fig. 3. HPLC chromatograms recorded during phenol decomposition under UV and with addition of H<sub>2</sub>O<sub>2</sub> on: (a) Fe–C500–TiO<sub>2</sub> and (b) Fe–TiO<sub>2</sub> photocatalysts.

Table 3

The characteristics of Fe–TiO<sub>2</sub> and Fe–C–TiO<sub>2</sub> with residual carbon

HTT (°C)	Sample code	BET (m <sup>2</sup> /g)	Carbon (mass%)	Fe <sup>3+</sup> (mass%)	Fe <sup>2+</sup> (mass%)	Phases by XRD (A, anatase; R, rutile)
400	Fe–C400–TiO <sub>2</sub>	146	3.3	0.37	0.77	A
500	Fe–C500–TiO <sub>2</sub>	72	2.4	0.96	0.70	A, FeTiO <sub>3</sub>
600	Fe–C600–TiO <sub>2</sub>	28	0.9	0.07	0.33	A, FeTiO <sub>3</sub>
700	Fe–C700–TiO <sub>2</sub>	7.5	0.3	–	–	A, R, FeTiO <sub>3</sub>
800	Fe–C800–TiO <sub>2</sub>	3	0.3	0.06	0.09	R, FeTiO <sub>3</sub>
900	Fe–C900–TiO <sub>2</sub>	2.5	0.2	0.16	0.08	R, FeTiO <sub>3</sub>
550	Fe–TiO <sub>2</sub>	55	0	1.3	0.6	A, FeTiO <sub>3</sub>

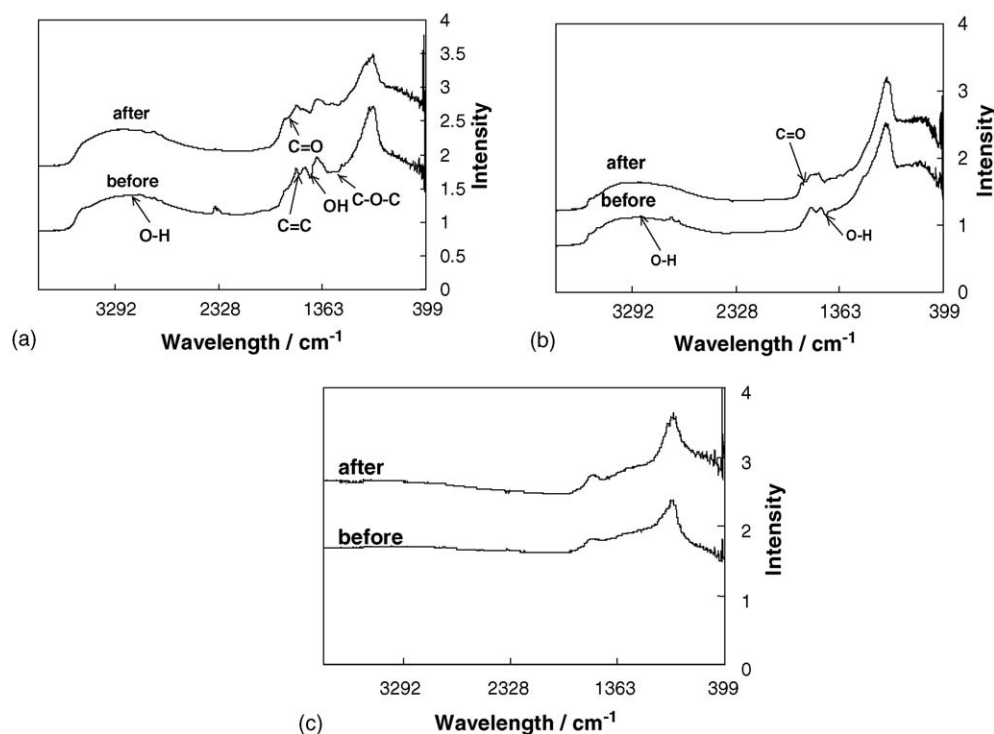


Fig. 4. FT-IR spectra of: (a) Fe–PET400–TiO<sub>2</sub>, (b) Fe–TiO<sub>2</sub> and (c) Fe–PET700–TiO<sub>2</sub> before and after 2 h of phenol decomposition under UV with addition of H<sub>2</sub>O<sub>2</sub>.

The highest rate of phenol decomposition was observed on the Fe–C500–TiO<sub>2</sub> sample, which had the highest content of iron. Although, the content of iron in Fe–TiO<sub>2</sub> was also high, the phenol decomposition was going slowly, even much slower than on Fe–C–TiO<sub>2</sub> photocatalysts having only rutile phase (Fe–C800–TiO<sub>2</sub> and Fe–C900–TiO<sub>2</sub>). It could be explained by the different mechanism of phenol decomposition. In Fig. 3, the chromatograms taken at different time of phenol decomposition under UV irradiation and with addition of H<sub>2</sub>O<sub>2</sub> on Fe–TiO<sub>2</sub> and Fe–C500–TiO<sub>2</sub> photocatalysts are presented.

It can be observed that phenol decomposition on Fe–C500–TiO<sub>2</sub> proceeds progressively, phenol is very quickly converted to HQ, catechol, BQ and some unidentified short-chain products (peak observed at the retention time of 1.5 min), then ring products are decomposed completely after 2 h of UV irradiation and amount of short chain products decreases, they are completely decomposed after 5 h. On Fe–TiO<sub>2</sub> sample, however, phenol is also converted to HQ, catechol and short chain products (no BQ is observed), the decomposition is going slowly with no selectivity,

all the organic compounds are decomposed gradually with time of UV irradiation, but also the highest concentration of HQ and catechol is observed after 2 h of UV irradiation.

Some FT-IR measurements of the chemical surface of Fe–C–TiO<sub>2</sub> and Fe–TiO<sub>2</sub> photocatalysts were performed before and after UV irradiation in the phenol solution with the addition of H<sub>2</sub>O<sub>2</sub>. The results are presented in Fig. 4.

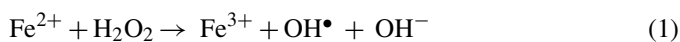
The chemical surface of Fe–PET400–TiO<sub>2</sub> photocatalyst changed after 2 h of phenol decomposition under UV with H<sub>2</sub>O<sub>2</sub>, some O–H and C=O bands reasonably attributed to hydroquinones and benzoquinone were formed (O–H band became wider), whereas on Fe–TiO<sub>2</sub> only C=O band appeared, which could be attributed to benzoquinone, and no change of the chemical surface of Fe–PET700–TiO<sub>2</sub> photocatalyst was observed.

#### 4. Discussion

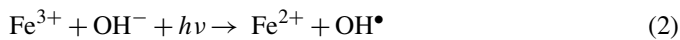
In the photo-Fenton process, the degradation of organic compounds proceeds by the formation of OH• radicals due to the



oxidation and reduction of iron. In the presence of  $\text{Fe}^{2+}$  and  $\text{H}_2\text{O}_2$ , the following reaction occurs:



Iron can be reduced in the presence of UV light and hydroxyl ions, according to the reaction:



Reaction (2) is less efficient than reaction (1), so when the iron exists only in the form of  $\text{Fe}^{3+}$ , then the process of decomposition is slowed down until buildup of  $\text{Fe}^{2+}$  [12].

From FT-IR measurements it could be concluded that formed hydroquinones, which were adsorbed on the surface of  $\text{Fe-C-TiO}_2$  photocatalyst during phenol decomposition played important role in the photo-Fenton reaction and could result in the acceleration of the phenol decomposition. Therefore, the contact of phenol molecules with the photocatalyst surface is important, in case of  $\text{Fe-C-TiO}_2$  photocatalysts with high carbon content which showed poor adsorption of phenol, the process of phenol decomposition was accelerated in the beginning, because of the oxidation of  $\text{Fe}^{2+}$  in the presence of  $\text{H}_2\text{O}_2$ , and then was slowed down due to the slow reduction of  $\text{Fe}^{3+}$ , what could be explained by the poor contact of formed hydroquinones with iron (Fig. 1).

On  $\text{Fe-C-TiO}_2$  photocatalysts with the residue carbon, phenol decomposition under UV and with  $\text{H}_2\text{O}_2$  was going faster in case of the photocatalysts, which had the higher content of iron, especially  $\text{Fe}^{2+}$  (Fig. 2), the speed of phenol decomposition was determined mostly by the occurring photo-Fenton reactions.

On  $\text{Fe-TiO}_2$  photocatalyst decomposition of phenol under UV with  $\text{H}_2\text{O}_2$  was accelerated in comparison with pristine  $\text{TiO}_2$ , however was going much slower than on  $\text{Fe-C-TiO}_2$  photocatalysts, as shown in Fig. 2. In that case phenol was decomposed by the radical reaction, which was not selective for phenol. Therefore, the presence of carbon in  $\text{Fe-C-TiO}_2$  photocatalysts seems to be important, because on  $\text{Fe-C-TiO}_2$  photocatalysts the phenol conversion was going quickly to ring products, which took part in the further mineralization of phenol.

## 5. Conclusions

High rate of phenol decomposition was obtained on  $\text{Fe-C-TiO}_2$  photocatalyst under UV irradiation with addition of small amount of  $\text{H}_2\text{O}_2$  to the phenol solution, because some photo-Fenton reactions worked for phenol decomposition. Decomposition of phenol on  $\text{Fe-C-TiO}_2$  photocatalysts proceeded mainly by the photo-Fenton process, oxidation of  $\text{Fe}^{2+}$  to  $\text{Fe}^{3+}$  in the presence of  $\text{H}_2\text{O}_2$  and the reduction of  $\text{Fe}^{3+}$  under UV and in the presence of some hydroquinones. It was proved that the rate of phenol decomposition was governed by

two factors, high content of iron in the photocatalyst (with a good balance between  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$ ), and a good contact of phenol molecules with the active iron species. In case of the high content of carbon (7.5–15.5 mass%) in  $\text{Fe-C-TiO}_2$  photocatalysts (comes from the carbonization of PET and oxalate), adsorption of phenol onto the surface of  $\text{Fe-C-TiO}_2$  is important to facilitate contact of phenol molecules with the active species formed during excitation of semiconductor and photo-Fenton reactions. In case of the low content of carbon in  $\text{Fe-C-TiO}_2$  photocatalysts (0.2–3.3 mass%), the rate of phenol decomposition is higher for the higher content of the active iron in the photocatalyst. On  $\text{Fe-TiO}_2$  photocatalyst, decomposition of phenol under UV irradiation and with addition of  $\text{H}_2\text{O}_2$  is going faster than on the pristine  $\text{TiO}_2$  due to the photo-Fenton reactions, but much slower than on the  $\text{Fe-C-TiO}_2$  photocatalysts modified by iron, which contain the carbon. On  $\text{TiO}_2$  and  $\text{Fe-TiO}_2$  photocatalysts phenol is decomposed by the radical reaction, which is not selective for phenol decomposition. From those measurements, it was proved that phenol decomposition is much favored by the complex reactions of phenol oxidation and it could be realized by photo-Fenton reactions with using  $\text{Fe-C-TiO}_2$  photocatalysts.

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