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## TITANIA-ASSISTED PHOTOCATALYTIC DECOMPOSITION OF TRITON X-100 DETERGENT IN AQUEOUS SOLUTION

Photocatalytic oxidation of nonionic surfactants (Tritons) taking place on TiO<sub>2</sub> surface is very efficient process. The shortening of polyoxyethylene chains and the splitting of phenyl group occur as a result of hydroxyl radical attack on Tritons molecules. The highest yield of Triton X-100 photooxidation found for the titania (TiO<sub>2</sub>) loading of 1.5–2 g/dm<sup>3</sup> strongly depends on the UV lamp applied – the kind of UV radiation and its intensity. The character of photooxidation was the same in the range of surfactant concentration below and above CMC characteristic of Triton X-100. The titania photocatalyst was not subjected to deactivation and could effectively be used many times.

### 1. INTRODUCTION

Both nonionic and ionic surfactants in aqueous solutions are excessively used in many formulations applied in various industrial areas such as detergent industry, agrochemistry, cosmetics, textiles and metallurgy. Two main groups of nonionic surfactants, i.e., alkylophenol polyetoxylates (APE) and alkylalcohols, demonstrate very useful properties, simplicity and low cost of production. Worldwide production of APE surfactants is  $5 \times 10^5$  tons per year [1] and they are unavoidably present in municipal sewage and industrial wastes.

Surfactants being the common pollutant of sewage belong to the chemical compounds which are hardly removable by classical physicochemical and biological methods. Because the APE surfactants display a low biodegradability and high toxicity for biologically active deposits their pretreatment should consist in an efficient oxidation. One of the advanced oxidation processes (AOPs) is an effective oxidation combined with the use of photocatalysts. The pretreated wastewater can be finally purified by fast, efficient and economically accepted biological process. AOP is applied to drinking water, sewage and

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air treatment in order to remove hardly degradable compounds. This method is based on the simultaneous action of combined oxidation agents (such as ozone, hydrogen peroxide, ultraviolet radiation) and aqueous suspensions of semiconductors producing very reactive oxidizing agents, i.e. hydroxyl radicals ( $\bullet\text{OH}$ ). The main advantages of AOP are as follows: high yield, fast reaction rate and low selectivity of action leading to nontoxic end products. Radical reactions can be applied to decomposition of many different chemical compounds, e.g. phenols, dyes, pesticides and surfactants [2]–[5].

The methods that are based on AOP with UV radiation can be divided into two categories [6]:

- homogeneous process – UV photolysis of  $\text{H}_2\text{O}_2$  and  $\text{O}_3$  or compounds generating free OH radicals,
- heterogeneous process based mainly on combined action of UV radiation and  $\text{TiO}_2$  (anatase).

Classification of AOPs according to reaction phase is presented in the table.

Table

Advanced oxidation processes [6]

Phase	Radiation	Process
Homogeneous	with radiation	$\text{O}_3/\text{UV}$
		$\text{H}_2\text{O}_2/\text{UV}$
		electron beam
		ultrasonic radiation
		$\text{H}_2\text{O}_2/\text{ultrasonic radiation}$
		$\text{UV}/\text{ultrasonic radiation}$
	without radiation	$\text{H}_2\text{O}_2/\text{Fe}^{2+}/\text{UV}$ (photo-Fenton)
		$\text{O}_3/\text{H}_2\text{O}_2$
		$\text{O}_3/\text{OH}^-$
Heterogeneous	with radiation	$\text{H}_2\text{O}_2/\text{Fe}^{2+}$ (Fenton reaction)
		$\text{TiO}_2/\text{O}_2/\text{UV}$
	without radiation	$\text{TiO}_2/\text{H}_2\text{O}_2/\text{UV}$
		electro-Fenton reaction

$\text{TiO}_2$ -assisted photocatalytic AOPs are based on the exposition of  $\text{TiO}_2$  surface to the radiation whose minimum energy 3.2 eV photon ( $h\nu$ ) can transfer an electron ( $e_{cb}^-$ ) from the valence band of crystal to the conduction band leaving an electron hole ( $h_{vb}^+$ ) (figure 1 [7]). This energy is equivalent to the photons of 388 nm wavelength. The activation of  $\text{TiO}_2$  takes place in the radiation range of 300–388 nm [7], [8]. Both the holes of valence band and the electrons of conduction band can recombine in a crystal lattice giving fluorescence and heat emission effects. They can also migrate on the catalyst surface, where they can be involved in redox reactions with adsorbed water molecules, hydroxyl groups  $\text{OH}^-$ , oxygen atoms and molecules of organic compounds.

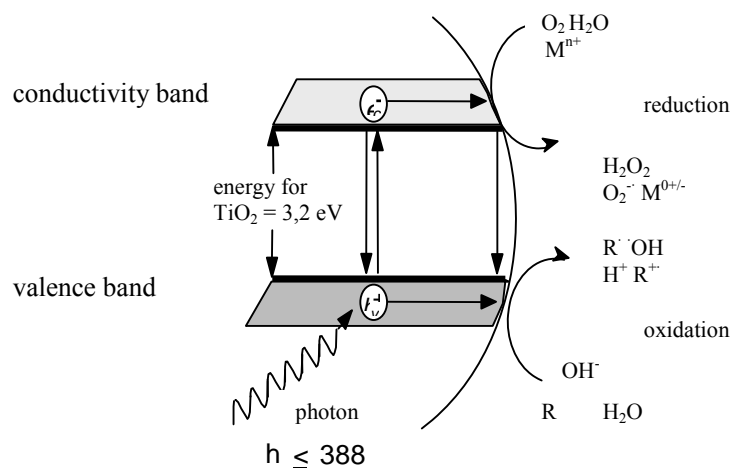
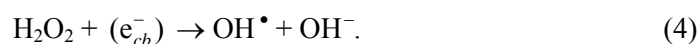
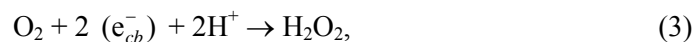


Fig. 1. Scheme of  $\text{TiO}_2$  – UV version of AOP process

The hydroxyl groups are produced as a result of oxidation between electron hole ( $h_{vb}^-$ ) and  $\text{H}_2\text{O}$  molecule or  $\text{OH}^-$  group:



Electron ( $e_{cb}^-$ ) reacts with adsorbed oxygen (aq) molecule to form  $\text{O}_2^-$  ion, which can form additionally  $\text{H}_2\text{O}_2$  and  $\text{HO}^\bullet$ :

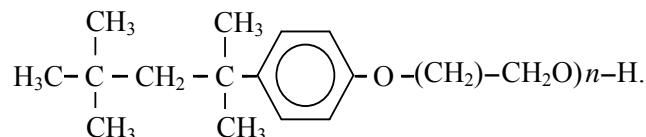


In lower pH range, hydrogen atoms and molecules can be produced [7], [9]–[10].

## 2. EXPERIMENTAL

### 2.1. MATERIALS

The aqueous solutions of polyoxyethylated alkylophenols known as commercial surfactant Triton X-100 were degraded in AOP. Triton is the product of condensing ethylene oxide and *p*-1,1,3,3 tertamethylbutylphenol (Fluka Chemie A.g.) and a mixture of oligomers with characteristic distribution at  $n = 9.5$ :



Titanium oxide (Degussa P25) was applied as photocatalyst whose surface area reaches 49 m<sup>2</sup>/g.

## 2.2. APPARATUS AND PROCEDURE

Photooxidation and photocatalytic processes were run in commercial photoreactor (Heraeus Laboratory, UV Reactor 451 08 261) that consisted of 1000-cm<sup>3</sup> glass vessel equipped with UV lamp (150 W or 8 W) being placed centrally in a Quartz lining and with magnetic rotor. Oxygen was delivered to the solution via a special tube at a constant volume rate of 10 dm<sup>3</sup>/h. Reaction temperature usually approached 20 °C. The known dose of the photocatalyst was added to the aqueous solution of Triton X-100 before the experiment. The of reaction lasted 5 hours. The light intensity was determined by FeK<sub>3</sub>(C<sub>2</sub>O<sub>4</sub>)<sub>3</sub>·(H<sub>2</sub>O)<sub>3</sub> actinometer. In the case where 8-W lamp emitted light in the range of 254–578 nm, intensity of light absorbed by aqueous solvent was estimated to be 1.249·10<sup>20</sup> photons/min dm<sup>3</sup> which corresponds to the energy consumption equal to 4.532·10<sup>20</sup> eV/min dm<sup>3</sup> or 72.5 J/min dm<sup>3</sup>. In the case of 150 W lamp, the intensity of light absorption was 1.91·10<sup>21</sup> photon/min dm<sup>3</sup> which is equivalent to the energy uptake of 5.81·10<sup>21</sup> eV/min dm<sup>3</sup> or 931·J/min dm<sup>3</sup>.

## 2.3. ANALYTICAL METHODS

The oxidation of surfactants was carried out in the system with photocatalyst as a suspension. After reaction the solution was filtered in order to analyze the samples. The reaction run was monitored spectrophotometrically (Hewlett Packard, spectrophotometer model HP 8542; absorbance was measured at 224 nm), polarographically (measurements of total organic carbon (TOC) (5000 Shimadzu)) and by chemical and biochemical oxygen demand (COD and BOD) methods. Also the high-resolution liquid chromatography (HRLC) method was applied to follow the composition of individual detergents. The process optimization was performed taking into account the following parameters: concentration and type of surfactant, the dose of surfactant, the mass of photocatalyst, pH of solution, the radiation intensity, the time of reaction and the possibility of reusing a catalyst, supportive action of O<sub>3</sub>, H<sub>2</sub>O<sub>2</sub> and UV. All analytical procedures were carried out according to Polish Standards.

### 3. RESULTS

The investigations of  $\text{TiO}_2$ -assisted UV photocatalytic oxidation of aqueous solutions of Triton X-100 surfactant were carried out applying different doses of  $\text{TiO}_2$ , different concentrations of Triton X-100 and two lamps of UV radiation. All experiments were repeated at least once.

#### 3.1. $\text{TiO}_2$ PHOTOCATALYST DOSE

Six different  $\text{TiO}_2$  suspensions were applied, i.e. 0.25, 0.5; 1.0; 1.5, 2.0 and 3.0 g per 1  $\text{dm}^3$  of Triton X-100 aqueous solution of the concentration of 120  $\text{mg}/\text{dm}^3$  ( $1.92 \times 10^{-1} \text{ mmol}/\text{dm}^3$ ). The use of a higher  $\text{TiO}_2$  dose was rather undesirable taking into account the efficiency of UV photocatalytic system. In the experiments, 150-W ultraviolet lamp was used. The influence of photooxidation time on absorbance ratio ( $A/A_0$ ) of Triton X-100 solution for six different doses of  $\text{TiO}_2$  is presented in figure 2. The insert shows the reduction of absorbance ratio versus the reaction time and  $\text{TiO}_2$  dose. The changes in COD, depending on  $\text{TiO}_2$  doses (0.5–3.0 g of  $\text{TiO}_2/\text{dm}^3$ ),

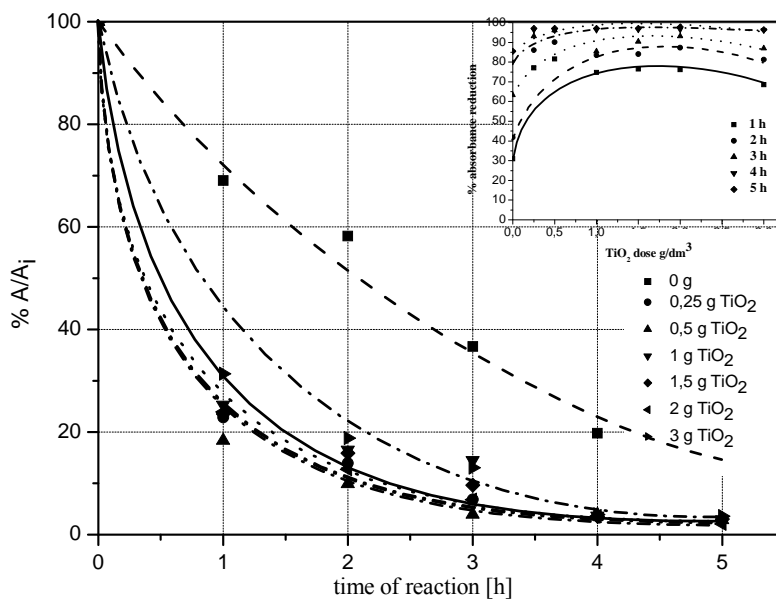


Fig. 2. Changes of the absorbance ratio  $A/A_0$  in the function of reaction time during photocatalytic oxidation of aqueous solution of Triton X-100 at different doses of  $\text{TiO}_2$  (0.25–3.0  $\text{g}/\text{dm}^3$ ). The insert illustrates reduction of solution absorbance, depending on  $\text{TiO}_2$  dose, for 1, 2, 3, 4 and 5 hours of reaction.

Experiments were performed with 150-W UV lamp

are presented in figure 3. The insert illustrates reduction of COD value in function of  $\text{TiO}_2$  dose for 3 and 5 hours of oxidation time.

For the sake of comparison the experiment without photocatalyst is presented. The insert illustrates COD value versus  $\text{TiO}_2$  dose for 3 and 5 hours of reaction. Experiments were performed with 150-W UV lamp.

The results of photochemical oxidation of Triton X-100 solutions lacking  $\text{TiO}_2$  catalyst are presented in figures 2 and 3. One can take under consideration different effects of the solution exposition to UV radiation because absorption of light in the systems with and without catalyst suspension is rather incomparable. The evaluation of UV radiation absorbed during photooxidation of surfactant dissolved in water was presented in our paper [11]. In the case of  $\text{TiO}_2$  suspension, most of UV radiation is absorbed by titania particles and the contribution of surfactant molecules to light absorption is relatively small. Supposedly, the proportion of dispersed light is significant but difficult to precise determination. The results analogous to those presented in figures 2 and 3 were obtained also for 8-W UV lamp. The optimal dose of  $\text{TiO}_2$  was 1.5 g per 1  $\text{dm}^3$  of surfactant solution for both UV lamps (8 and 150 W). During 5-hour reaction the absorbance of Triton X-100 solution decreased by 75%, and the COD and TOC values decreased by 28% and 26%, respectively. Transparency of titania suspension decreased with an increase in  $\text{TiO}_2$  loading and the contribution of UV radiation to surfactant decomposition decreased.

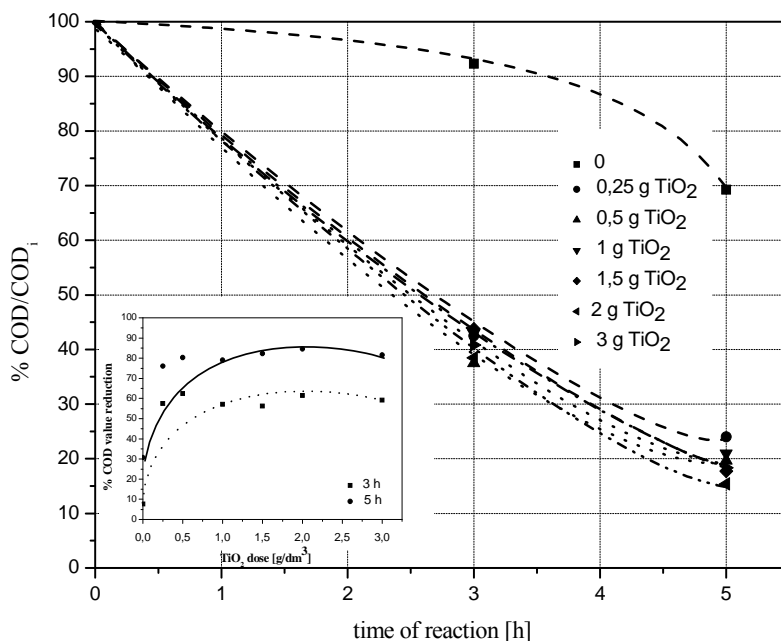


Fig. 3. Changes of COD values in the function of reaction time during photocatalytic oxidation of aqueous solution of Triton X-100 at different doses of  $\text{TiO}_2$  (0.25–3.0  $\text{g}/\text{dm}^3$ )

## 3.2. TRITON X-100 CONCENTRATION

Photocatalytic process was investigated at the Triton X-100 concentration ranging from  $40 \text{ mg/dm}^3$  ( $6.4 \times 10^{-2} \text{ mol/dm}^3$ ) to  $250 \text{ mg/dm}^3$  ( $4 \times 10^{-1} \text{ mol/dm}^3$ ). The critical micellar concentration (of aqueous solution) was about  $150 \text{ mg/dm}^3$  for Triton X-100. Thus, surfactant had the form of molecules ( $40, 80$  and  $120 \text{ mg/dm}^3$ ) and micellar species ( $160, 200$  and  $250 \text{ mg/dm}^3$ ). During 5-hour reaction the changes in solution absorbance  $A$  and COD values were measured. The amount of photocatalyst ( $\text{TiO}_2$ ) ranged from  $1.5$  to  $2 \text{ g/dm}^3$  and two UV lamps ( $8$  and  $150 \text{ W}$ ) were used. The absorbance and COD versus reaction time are presented in figures 4 and 5, respectively. Inserts show the reduction of absorbance and COD values, depending on Triton X-100 concentration after 1, 3 and 5 hours of photooxidation.

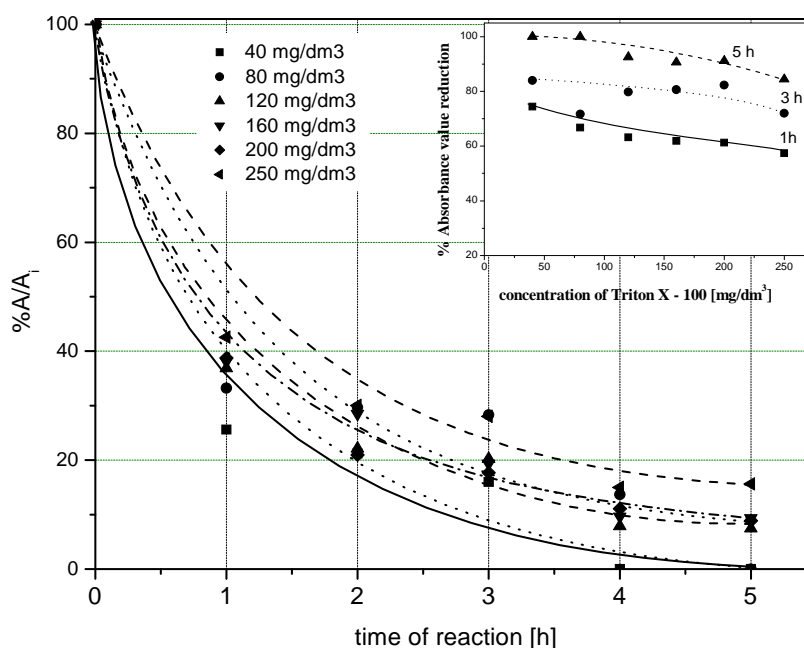


Fig. 4. Changes of the absorbance ratio  $A/A_0$  during photooxidation of aqueous solutions of Triton X-100 at initial concentration of surfactant in the range of  $40\text{--}250 \text{ g/dm}^3$ .

The insert illustrates reduction of solution absorbance, depending on the Triton X-100 concentration, for 1, 3 and 5 hours of reaction. Experiments were performed with 150-W UV lamp

It can be seen that the increase in Triton X-100 concentration results in lower COD and absorbance reduction by about 20 and 10%, respectively, at 40 and 250 mg of surfactant per one  $\text{dm}^3$  after 3 and 5 hours of reaction. In both cases, the solution exhibited neither molecular nor micellar character even in the vicinity of CMC characteristic of Triton X-100 aqueous solution.

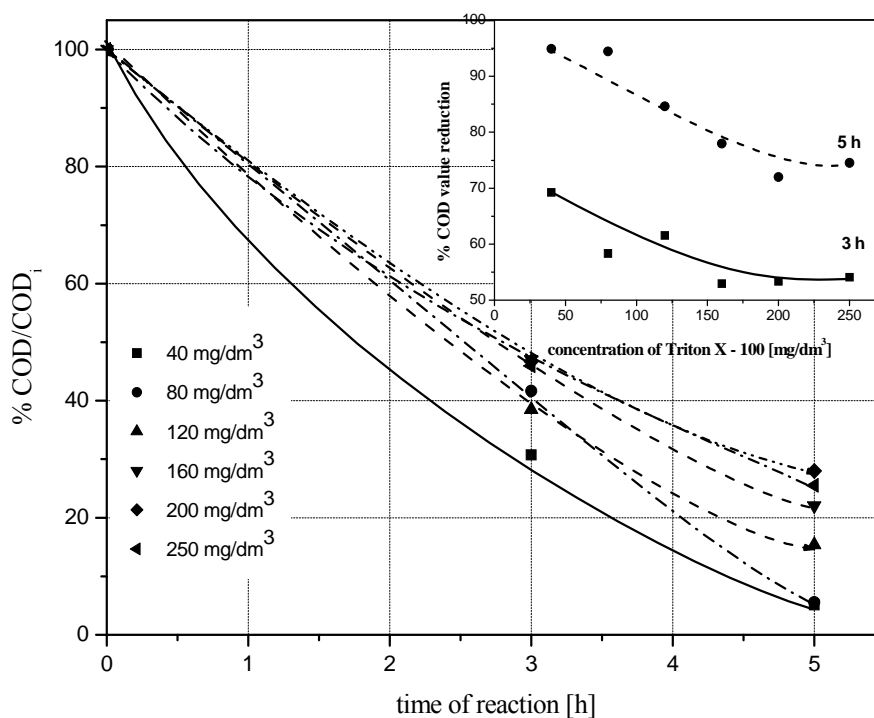


Fig. 5. Changes of COD values in the function of reaction time during photocatalytic oxidation of aqueous solutions of Triton X-100 at initial concentration of surfactant in the range of 40–250 g/dm<sup>3</sup>. The insert illustrates reduction of COD value, depending on Triton X-100 concentration, for 3 and 5 hours of reaction. Experiments were performed with 150-W UV lamp

### 3.3. UV RADIATION INTENSITY

The influence of UV radiation intensity (8- and 150-W lamps) on the decomposition efficiency of Triton X-100 aqueous solution is illustrated in figure 6 as  $A/A_0$  and  $COD/COD_0$  changes in the function of reaction time.

In the case of 8-W lamp emitting light in the range of 254–578 nm, the amount of the light absorbed by aqueous solvent was estimated to be  $1.249 \cdot 10^{20}$  photons/min dm<sup>3</sup> which corresponds to the energy consumption equal to  $4.532 \cdot 10^{20}$  eV/min dm<sup>3</sup> or 72.5 J/min dm<sup>3</sup>. For 150-W lamp the intensity of light absorption was  $1.91 \cdot 10^{21}$  photons/min dm<sup>3</sup> which is equivalent to energy uptake of  $5.81 \cdot 10^{21}$  eV/min dm<sup>3</sup> or 931 J/min dm<sup>3</sup>.

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$72.5 \text{ J/min dm}^3$ . For 150-W lamp the intensity of light absorption was  $1.91 \cdot 10^{21}$  photons/min  $\text{dm}^3$  which is equivalent to energy uptake of  $5.81 \cdot 10^{21}$  eV/min  $\text{dm}^3$  or  $931 \text{ J/min dm}^3$ .

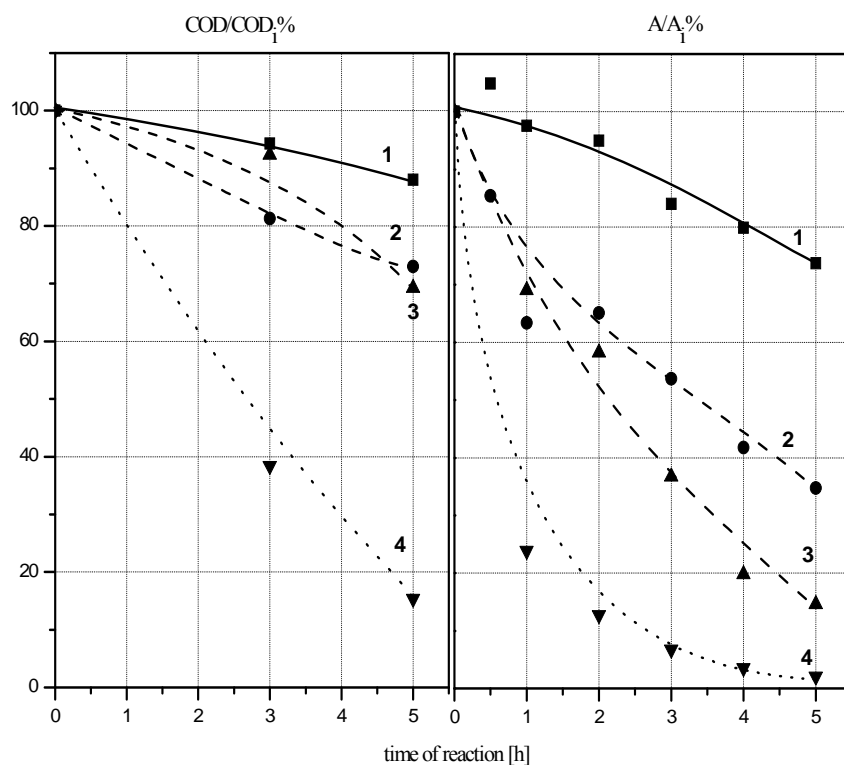


Fig. 6. The influence of photodecomposition parameters of water solution of Triton X-100 given as the  $\text{COD}/\text{COD}_0$  and  $A/A_0$  ratios versus reaction time: **1** – 8 W +  $\text{O}_2$ , **2** – 8 W +  $\text{O}_2$  +  $2 \text{ g/dm}^3 \text{ TiO}_2$ , **3** – 150 W +  $\text{O}_2$ , **4** – 150 W +  $\text{O}_2$  +  $2 \text{ g/dm}^3 \text{ TiO}_2$

Emission spectra of both UV lamps were different. The efficiencies of emitted light in the wavelength range of 250–350 nm were  $5.73 \times 10^{19}$  and  $50.5 \times 10^{19}$  photons/min  $\text{dm}^3$  for 8-W and 150-W lamps, respectively. At the wavelength of 254 nm the efficiencies were similar and close to  $4.85 \times 10^{19}$  and  $5.62 \times 10^{19}$  photons/min  $\text{dm}^3$ , respectively. Power ratio for both lamps was 18.75 which corresponds to the ratio of efficiencies in the entire range of emission spectrum. However, in the range of UV, this ratio is different and equal to about 9. Ultraviolet radiation is essential for photocatalytic decomposition of Triton X-100. That is the reason for low decrease of  $\text{COD}/\text{COD}_0$  and  $A/A_0$  ratios in the initial stages of decomposition.

### 3.4. REUSE OF TiO<sub>2</sub> PHOTOCATALYST

Additionally a sequence of triple reuse of the same sample of TiO<sub>2</sub> catalyst in 5-hour reaction cycle was performed and this cycle of experiments confirmed the same catalyst photoactivity. The sample of TiO<sub>2</sub> was separated from the solution after one reaction run and reused in next experiment. Practically the same changes of COD and absorbance values in successive experiments were measured.

## 4. DISCUSSION

Photocatalytic oxidation is a complex process. Three phases of TiO<sub>2</sub>-assisted photocatalytical process can be anticipated: adsorption of chemical compound on catalyst surface, oxidation of the compound adsorbed on the catalyst surface and simultaneously oxidation of compounds in aqueous solution. Usually the last process of oxidation can be treated as photooxidation because additionally the solution is strongly saturated with gaseous oxygen. In order to give us a deeper insight into mechanism of compound decomposition in aqueous solution, the adsorption of surfactants and their photodecomposition on titania surface should be estimated. Evaluation of percentage contribution of adsorption, photooxidation and TiO<sub>2</sub>-assisted photooxidation to the total decomposition process is presented in figure 7 as COD reduction obtained after 5 hours. Rather clear differences can emerge from these results. Triton X-100 adsorption on titania surface is relatively small. The contribution of photooxidation and TiO<sub>2</sub>-assisted photooxidation to decomposition are much higher and the highest, respectively. Photooxidation does not take place in the conditions exactly the same as those during catalytic photooxidation. Although the amount of the radiation absorbed can be slightly different but its main portion is dispersed on titania grains in TiO<sub>2</sub>-UV oxidation. And the adsorbed molecules of surfactant undergo oxidation on the catalyst surface or can desorb into the liquid phase. Despite this obvious limitation the photocatalytical process is dominant in Triton X-100 decomposition.

The optimum dose of TiO<sub>2</sub> in aqueous suspension, i.e. 1.5 g/dm<sup>3</sup>, is analogical to that used in decomposition of other compounds. For example, the dose of 1.0 g of TiO<sub>2</sub> per one dm<sup>3</sup> was used for oxidation of benzene, chlorobenzene, DDT, and the following acids: *p*-nitrobenzoic, *p*-aminobenzoic and *p*-chlorophenoxyacetic [12], [13], while 2-g dose of TiO<sub>2</sub>, for hexafluorobenzene oxidation [14]. Lower titania doses were applied to more diluted solutions of chemical compounds [15]. WANG [16] estimated 0.25 g/dm<sup>3</sup> as the optimum dose of TiO<sub>2</sub> to oxidize textile sewage and found 34% reduction of COD at pH = 3. CHUN [4] increased TiO<sub>2</sub> dose to 6.25 g/dm<sup>3</sup> to decompose aqueous solution of phenol using UV lamp with 200–300 nm spectral range of light emission.

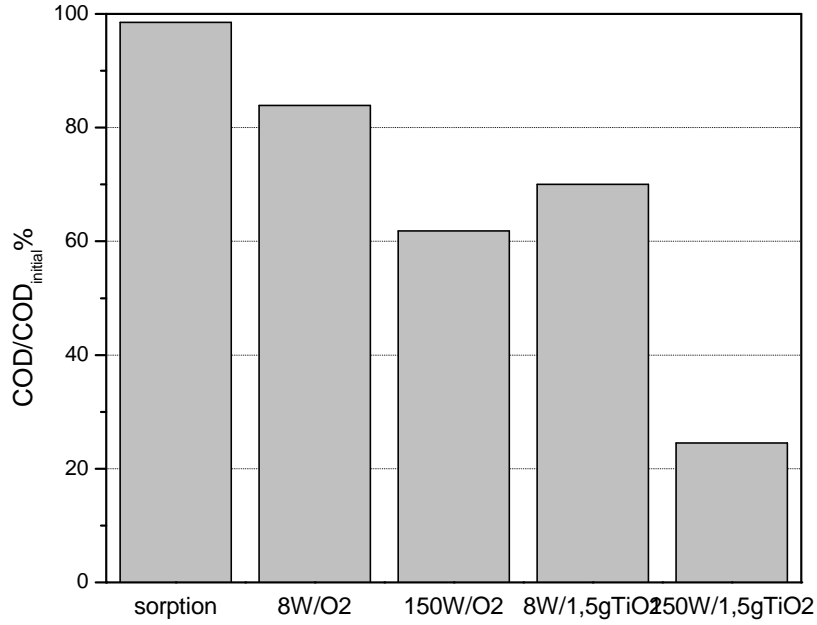


Fig. 7. Decrease in COD for Triton X-100 solution (120 mg/dm<sup>3</sup>) after 5 hours. TiO<sub>2</sub> dose of 1.5 g/dm<sup>3</sup>. Experiments were performed with 8- or 150-W UV lamps

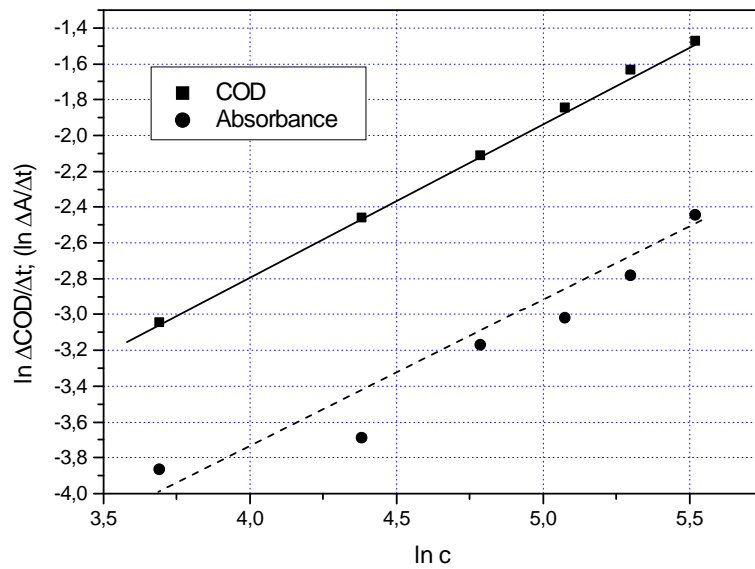


Fig. 8. The changes of initial ratios of  $\Delta\text{COD}/\Delta t$  and absorbance  $\Delta A/\Delta t$  in the function

of an initial Triton X-100 concentration

The influence of an initial concentration of Triton X-100 on its decomposition in the beginning of one-hour period is presented in figure 8 as double logarithmic straight lines. Based on these lines the exponential factor in equation can be calculated. According to literature data the first-order reaction at the rate constant of about  $10^{-3} \text{ s}^{-1}$  was postulated for photodecomposition of benzene, chlorobenzene, diazo dyes [17], whereas in some cases the second-order reaction was also assumed [18]. The rate of photocatalytic decomposition is strongly depended on UV lamp power and spectral emission range. WANG [16] confirmed the influence of the light intensity applied on the degree of impurities removal. The same conclusion that photodegradation of sodium dodecyl sulphate was proportional to the intensity of the UV radiation applied was drawn by LEA [19].

Triplicate use of the same  $\text{TiO}_2$  catalyst without any noticeable decrease in its activity is very practical if its industrial applications are taken into account. Similar results reported by ARANA [2] referred to phenol photodecomposition (about 30% reduction of COD) in five-time cycle of  $\text{TiO}_2$  reuse. The catalyst changed its colour from white to brown.

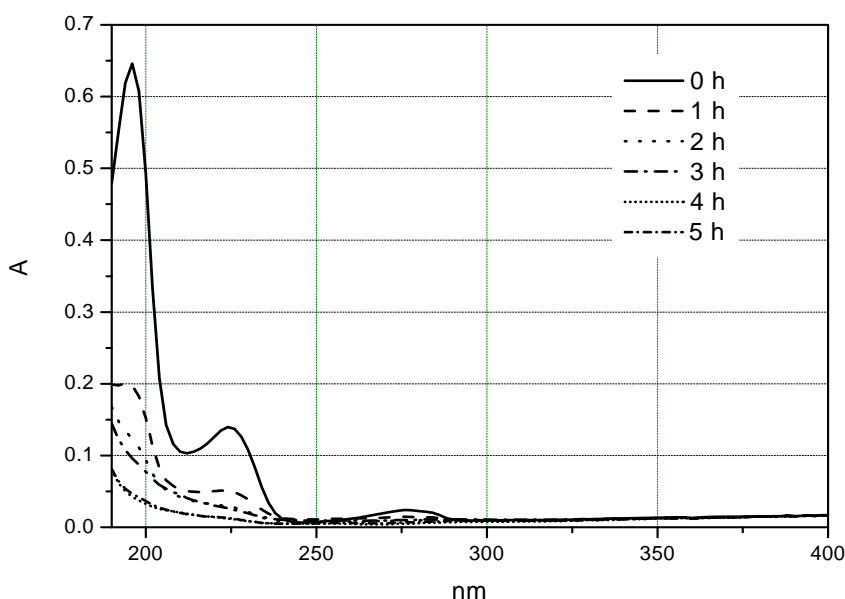


Fig. 9. UV-VIS spectra of aqueous solution of Triton X-100 ( $120 \text{ mg/dm}^3$ ) versus the time of photocatalytic oxidation.  $\text{TiO}_2$  dose of  $2 \text{ g/dm}^3$  and 150-W UV lamp

The examples of additional UV-VIS and HPLC measurements are given in figures

9 and 10, respectively. Interpretation of these and other findings leads to a general conclusion that radicals of hydroxyl groups being the major oxidation agent lead both to the shortening of oxyethyl chains and the opening of benzene ring in Triton molecules. The changes of UV-VIS spectra before and after Triton X-100 decomposition testify to the changes in its isomeric composition and distribution. The attack of hydroxyl radicals has probably statistical character and the number of oxyethyl mers in the Triton X-100 chain decrease from the initial value of about 9.5 to 2–4 groups after 5-hour photocatalytic oxidation.

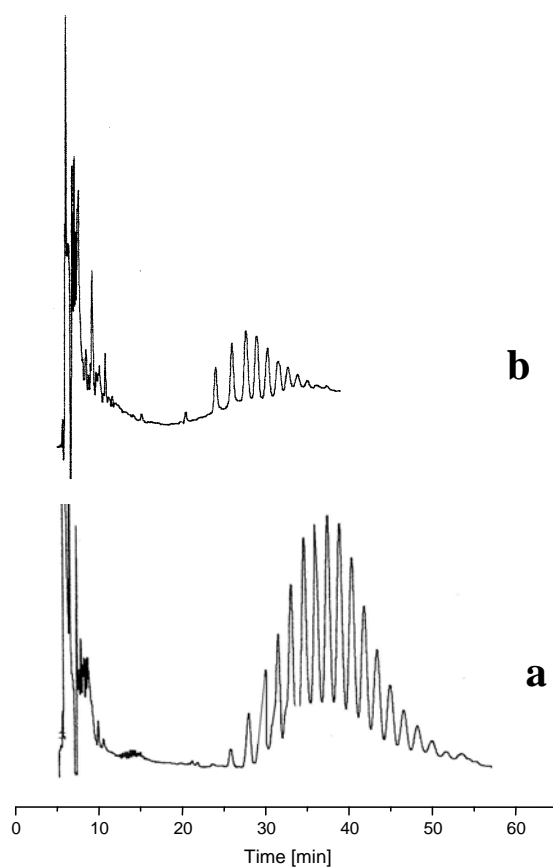


Fig. 10. The example of HPLC chromatograms of aqueous solution of Triton X-100 ( $120 \text{ mg/dm}^3$ ): at the beginning (a) and after 1-h photocatalytic oxidation (b).  $\text{TiO}_2$  dose of  $2 \text{ g/dm}^3$  and 8-W UV lamp

HPLC spectral changes of the Triton X-100 solution seem to confirm parallel attack of hydroxyl radicals on aromatic ring. During the first hour of photocatalytic oxidation about 70% of area enveloped by chromatographic profile disappears, but the

character of spectra remains the same (see figure 10). In more advanced stages of decomposition, spectral changes can be attributed to qualitative changes of Triton molecules leading to various intermediate compounds like 1,1,3,3-tetramethylbutylphenol, but their concentration is relatively small. The attack of hydroxyl radicals on oxyethyl chains cannot be excluded. The presence of branched nonylphenols should be confirmed experimentally.

## 5. CONCLUSION

Photocatalytic oxidation of nonionic surfactants (Tritons) taking place on TiO<sub>2</sub> surface is very efficient process. The shortening of polyoxyethylene chains and the splitting of phenyl group occur as a result of hydroxyl radical attack on Triton molecules. The highest yield of the Triton X-100 photooxidation found for TiO<sub>2</sub> load ranging from 1.5 to 2 g/dm<sup>3</sup> greatly depends on the UV lamp applied, i.e. the kind of UV radiation and its intensity. The same character of photooxidation was found in the range of surfactant concentration below and above CMC characteristic of Triton X-100. Deactivation of photocatalyst was not observed and multiple reuse of TiO<sub>2</sub> photocatalyst without the loss of its activity is possible.

## ACKNOWLEDGEMENT

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#### FOTOKATALITYCZNY PROCES ROZKŁADU TRITONU X-100 W ROZTWORZE WODNYM Z UDZIAŁEM TiO<sub>2</sub>

Fotokatalityczne utlenianie trudno rozkładalnych niejonowych detergentów (Tritonów) na powierzchni ditlenku tytanu jest procesem złożonym. Katalitycznemu utlenianiu NSPC towarzyszy adsorpcja związku na powierzchni TiO<sub>2</sub> i fotoutlenianie pod wpływem światła UV i tlenu obecnego w roztworze. Rodniki hydroksylowe powodują zarówno skracanie łańcucha polioksyetylenowego, jak i rozrywanie pierścienia aromatycznego w cząsteczce Tritonu. Najwyższą wydajność procesu rozkładu Tritonu X-100 otrzymano dla dawki TiO<sub>2</sub> w zakresie 1.5–2 g/dm<sup>3</sup>. Jej wielkość była wyraźnie zależna od intensywności zastosowanej lampy UV. Wielokrotne użycie tego samego katalizatora nie spowodowało spadku efektywności reakcji.