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Pt/TiO$_2$-ASSISTED PHOTOCATALYTIC DECOMPOSITION OF TRITON X-100 DETERGENT IN AQUEOUS SOLUTION

Noble metals, usually platinum or palladium, supported on titania are commonly used to improve the effectiveness of photocatalyst in decomposing aqueous solutions of organic compounds. 0.5 and 2% platinum supported on TiO$_2$ (P25 – Degussa) and 10% TiO$_2$ supported on SiO$_2$ appeared to be very active in powerful oxidation of Tritons, nonionic surfactants, dissolved in water. A platinum-promoted photocatalyst was by about 20% more active than TiO$_2$ alone. Titania supported on silica makes Pt/TiO$_2$/SiO$_2$ photocatalyst reusable because it can be very easily separated from aqueous solution after decomposition process

1. INTRODUCTION

An effective, fast and economically reasonable sewage purification leads to several-stages technology combining the chemical, physical and biological methods. Surfactants being the common contaminants of sewage are not regarded as biologically very toxic chemical compounds, but as hardly removable by classical, both physicochemical and biological methods. When the concentration of surfactants is higher than several tens of mg/dm^3 an aqueous solution shows negative interaction leading to a substantial decrease in surface energy which is not suitable for biological sewage purification. Tremendous amounts of foam on the surface of sewage make the access of air oxygen very difficult and the removal of active biological solid and sparingly soluble and cancerogenic chemical compounds much easier [1]. Among all the surfactants produced those known as hard detergents are only weakly biodegradable and do not selfdecompose in storage reservoir of water. Thus, their chemical oxidation in order to quench foam is a prerequisite for further water purification.

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One of the most efficient purification methods is the oxidation process combined with the use of photocatalysts. This method is based on a modern chemical technology of sewage and water treatment known as AOP (advanced oxidation process). In such a process, hydroxyl radicals (HO$^\bullet$) that evolve in different ways are exceedingly reactive. If TiO$_2$ surface is exposed to the radiation of minimum energy of 3.2 eV, photon (hv) can transfer an electron (e_{cb}^-) from the valence band of crystal to the conduction band leaving an electron hole (h_{vb}^+). This energy is equivalent to energy of the photons with the wavelength of 388 nm. The activation of TiO$_2$ takes place in the radiation range of 300–388 nm. Both, the holes of valence band and the electrons of conduction band can recombine in crystal network, resulting in fluorescence and heat evolution effects and they can migrate on a catalyst surface, where they can be involved in redox reactions with adsorbed water molecules, hydroxyl groups (OH$^-$), oxygen atoms and the molecules of organic compounds. The hydroxyl groups are produced as a result of oxidation between the hole (h_{vb}^+) and H$_2$O molecule or OH group. Electron can react with adsorbed O$_2$(aq) generating O$_2^-$, which additionally can give H$_2$O$_2$ and HO$^\bullet$ [2], [3].

The pretreated wastewater can be entirely purified in fast, efficient and profitable biological process. Many compounds, e.g., phenols, chlorobenzene, chloromethanes, pesticides, dyes and different type detergents, may be subjected to photocatalytic oxidation [4]–[7]. Most papers are focused on the parameters of photocatalytic optimization. The parameters that delay applying this process are: rather low efficiency of photon-induced hydroxyl radicals generation and the necessity of ultraviolet radiation. More effective photocatalysts working in the range of visible light are necessary as they make an industrial application of the sun or an inexpensive lamp radiation economically acceptable.

Up till now the most of laboratory investigations were performed in low-capacity reactors (about 1 dm^3) with UV lamps of several hunderds wats. TiO$_2$ (P25 Degussa) photocatalyst was added to form the suspension with aqueous solution which was constantly mixed by the bubles of oxygen stream or rotator. Special methods of TiO$_2$ separation are usually required: microfiltration or centrifuging; their industrial application increases the costs of the treatment process. In order to solve this problem, TiO$_2$ can be dispersed on the great surface of silica powder particles, spheres or interior surface of teflon and glassy fibres [8], [9]. Platinum-promoted photocatalysts are more active than TiO$_2$ alone. Degussa 5% Pt/TiO$_2$ catalyst was 2.1 and 1.4 times more active than TiO$_2$ alone in dichloracetic acid and p-chlorophenol decomposition, respectively. In the same conditions also Sachtleben Hombikat 0.2% Pt/TiO$_2$ UV 100 catalyst was 1.4 and 1.2 times more active [10]. 0.1–10% palladium photocatalysts appeared to be active in oxidation of many compounds [11]. SIEMON et al. [12] used 0.2–0.5% Pt/TiO$_2$ catalysts for the reduction of Cr ions and EDTA decomposition.

SURI et al. [13] found that destruction of organic contaminants was 6 times higher, whereas ZHANG et al. [6] claimed that degradation of pollutants was 16 times higher in the presence of $Pt/TiO_2/SiO_2$ catalyst.

2. EXPERIMENTAL

2.1. COMPOUNDS SUBJECTED TO DEGRADATION

The aqueous solutions of ethyloetoxylated alkylophenol, known as commercial surfactant Triton X-100, were degraded in AOP. Triton is a product of ethylene oxide condensation with *p*-1,1,3,3 tertmethylobuthylophenol (Fluka Chemie A.g.) being a mixture of oligomers with characteristic chain length distribution for $n = 9.5$.

The investigations presented in this paper are focused on the activity of $Pt/TiO_2/SiO_2$ photocatalyst system in the oxidation of Tritons, nonionic surfactants dissolved in water. The problem of catalyst separation and its reuse optimization is examined as well. The following catalysts were used:

Titanium dioxide – TiO_2 (P25 – Degussa).

Platinum-promoted titania – 0.5% Pt/TiO_2, 2% Pt/TiO_2.

Titania supported on silica – 10% TiO_2/SiO_2.

Platinum-promoted titania supported on silica – 0.5% Pt/ 10% TiO_2/SiO_2, 2% Pt/ 10% TiO_2/SiO_2.

2.2. CATALYST PREPARATION

Silica support was obtained by sol–gel method using tertetoxytitanium(IV) as an initial compound [14]. Titania supported on silica (10% TiO_2/SiO_2) was prepared by TiO_2 deposition on silica suspended in hexane solution of titanium isopropanol as a result of the hydrolysis of the latter in argon atmosphere and left for 48 hours at room temperature. Then solvent was removed in vacuum and the catalyst was dried at 120 °C for 4 h and finally calcined for 3 hours at 400 °C in oxygen stream.

Platinum-supported catalysts, i.e., Pt/TiO$_2$ and Pt/10% TiO$_2$/SiO$_2$, were prepared by the support impregnation in aqueous solution of H$_2$PtCl$_6$. After being kept for 24 hours at room temperature the catalysts were dried at 120 ºC for 4 hours and finally calcined for 3 hours at 200 ºC in oxygen stream [15]. The photocatalyst surface area and porosity measurements were carried out by BET (N$_2$) method.

2.3. TEMPERATURE– PROGRAMMED REDUCTION IN HYDROGEN (TPR$_{H2}$)

Temperature-programmed reduction (TPR$_{H2}$) and temperature-programmed oxidation (TPO) measurements were carried out in an AMI1 system from Altamira Instruments (USA) equipped with thermal conductivity detector (TCD) and/or MS quadrupole mass spectrometer. In TPR$_{H2}$ and TPO measurements, gaseous mixtures of 95% Ar–5% H$_2$ and 98% Ar–2% O$_2$, respectively, were used at the volume velocity of 60 cm^3 min^{-1} and linear rise in the temperature of 15 °C·min^{-1}. TPR$_{H2}$ measurements were carried out after fresh catalyst oxidation (O$_2$, 3 hours) at the oxidation temperature of 500, 700 and 900 °C.

2.4. ACTIVITY MEASUREMENTS AND ANALYTICAL PROCEDURE

Photooxidation and photocatalytic processes were carried out in photoreactor (Heraous) consisting of a glass vessel (volume of 1000 cm^3) equipped with UV150 W or 8 W lamp placed in a quartz lining and magnetic rotor. The solution was supplied with oxygen via a special tube, and its constant volume velocity of 10 dm^3 h^{-1} was kept. Temperature of reaction usually reached about 20 °C. The known amount of photocatalyst was added to aqueous solution of Triton X-100 before the experiment. The time of reaction ranged from 5 to 8 hours .

The oxidation of Triton X-100 aqueous solution was carried out in the system with the photocatalyst in the form of suspension. The course of reaction was controlled by spectrophotometeric method. The measurements of total organic carbon (TOC) were performed in 5000 Shimadzu device. The chemical and biochemical oxygen demand (COD and BOD) methods were also applied. A high-resolution liquid chromatography HRLC method was applied to follow the composition of individual detergents. The process was optimized based on the following parameters: the concentration and type of surfactant, the dose of surfactant, the photocatalyst mass, pH of solution, the radiation intensity, the reaction time and the possibility of reusing catalyst, a supportive action of O$_3$, H$_2$O$_2$ and UV. All analytical procedures were carried out according to Polish Standards.

3. RESULTS

3.1. SURFACE AREA

The influence of oxidation temperature (200–700 °C) on the results of surface area measurements is presented in the table. An increase in the oxidation temperature from 200 up to 700 °C results in a decrease in the surface area from about 50 to about 10 m^2/g, respectively. Platinum-supported catalysts have practically the same surface area as the supports themselves. A decrease of more than 10% in surface area is observed when SiO$_2$ is compared with SiO$_2$ covered with titania (10% TiO$_2$/SiO$_2$) which is a result of partial blocking of silica pores by titania. The diameter of pores that predominate decreases from 50 nm for SiO$_2$ to about 40 nm for 10% TiO$_2$/SiO$_2$.

Table

Photocatalyst surface area

Catalysts	Surface area (m^2/g)					
	Temperature of oxidation (°C)					
	200	300	400	500	600	700
TiO$_2$	50	45	42	31	11	9
0.5% Pt/TiO$_2$	49			30		9
2% Pt/TiO$_2$	49	43	40	29	10	10
5% Pt/TiO$_2$	40			25		8
SiO$_2$	426					
10% TiO$_2$/ SiO$_2$	382					
0.5% Pt/TiO$_2$/SiO$_2$	380					
2% Pt/TiO$_2$/SiO$_2$	363					
5% Pt/TiO$_2$/SiO$_2$	343					

3.2. TEMPERATURE-PROGRAMMED REDUCTION IN HYDROGEN

The influence of oxidation temperatures of 500, 700 and 900 °C on TPR$_{H2}$ profiles is presented in figures 1–4 for: TiO$_2$, 10% TiO$_2$/SiO$_2$, 0.5% Pt/TiO$_2$, 0.5% Pt/10% TiO$_2$/SiO$_2$ catalysts.

For TiO$_2$ alone the reduction effects are relatively small and appear in a broad temperature range, i.e., 200–900 °C (figure 1). An increase in the oxidation temperature from 500 to 700 and finally 900 °C does not infuence very much the reduction behaviour of TiO$_2$ and leads to rather unsubstantial decrease in TiO$_2$ reducibility caused by its sintering during the catalyst preoxidation at 700 and 900 °C, and the initial surface area of 50 m^2/g decreased to about 9 and 8 m^2/g, respectively.
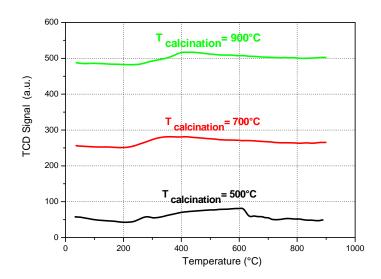
Fig. 1. The influence of temperature of oxidation (4 h, O_2) on TPR_{H2} profiles for TiO_2

Titania belongs to nonstoichiometric and partly reducible oxides formulated as TiO_{2-x}, where $0.5 \geq x \geq 0$ greatly depends on the atmosphere and treatment temperature. In air, at 870 °C the phase transformation from anatase into rutile takes place [16]. Commercial titania P25 usually contains 20% of anatase and 80% of rutile. Hovewer, in hydrogen atmosphere, the temperature of phase transformation of the Pt/TiO_2 catalyst is considerably lower.
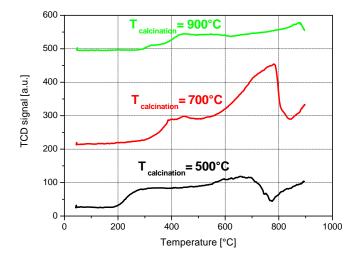

Fig. 2. The influence of temperature of oxidation (4 h, O_2) on TPR_{H2} profiles for
10% TiO_2/SiO_2 catalyst

The development of TiO$_2$ surface area by its dispersion on silica surface increases its reduction effects. After oxidation at 500 °C the reduction effects in the temperature range from 200 to 700 °C without any visible reduction maximum are presented in figure 2. The considerable changes in reduction behaviour can be noticed after oxidation at 700 °C – the beginning of reduction at higher temperature (350 °C) and well-defined maximum at about 800 °C. The amount of the hydrogen consumed is considerably higher than that after oxidation at 500 °C. The oxidation at still higher temperature, i.e., 900 °C, leads to a substantial decrease in reduction effects which is attributed to the sample sintering and phase transformation.
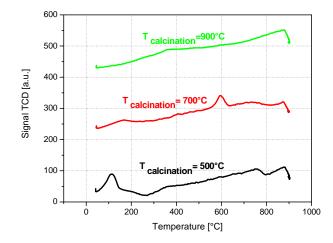
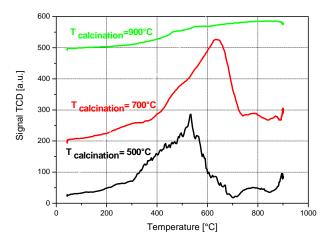Fig. 3. The influence of temperature of oxidation (4 h, O$_2$) on TPR$_{H2}$ profiles for 0.5% Pt/TiO$_2$ catalyst

Fig. 4. The influence of temperature of oxidation (4 h, O$_2$) on TPR$_{H2}$ profiles for 0.5% Pt/10% TiO$_2$/SiO$_2$ catalyst

Platinum (0.5% by weigh) supported on 10% TiO_2/SiO_2 surface increases considerably the consumption of hydrogen, especially after oxidation at 500 and 700 °C (see figure 4). In both cases, the reduction effect in the temperature range of 300–700 °C can be assigned to platinum promoting titania reduction, $TiO_2 + xH_2 \rightarrow TiO_{2-x}.+ xH_2O$. Oxidation at 900 °C leads to a substantial reduction of metal-support bridges Pt–O–Ti stabilized by chlorine present on titania surface and originating from H_2PtCl_6 [17]–[20]. TPR peaks characteristic of chlorine-stabilized platinum oxide phase were not observed. High-temperature (900 °C) oxidation of $Pt/TiO_2/SiO_2$ catalyst can lead to irreducible type of crystalline structure ($PtTiO_3$) strongly dispersed on the catalyst surface or incorporated into bulk catalyst [20].

Different character of reduction behaviour is shown in figure 3 for 0.5% Pt/ TiO_2 catalyst. After oxidation at 500 °C the low temperature peak in the range of 50–150 °C is attributed to chlorine-stabilized platinum oxide phase (PtO_xCl_y). The increase in the oxidation temperature to 700 °C gives the additional reduction effect with the maximum at about 600 °C. TPR profiles for 2% Pt/ TiO_2 and 2% $Pt/TiO_2/SiO_2$ catalysts appeared to be analogous to those that represent the catalyst containing 0.5% Pt.

3.3. PHOTOCATALYTIC OXIDATION OF TRITON X-100

During photocatalytic oxidation of the aqueous solution of Triton X-100 the following parameters were measured: solution pH, UV absorbance ratio (A/A_0), total organic carbon (TOC), chemical and biochemical oxygen demand (COD and BOD_5). In order to remove TiO_2, the solution was filtered before analysis.
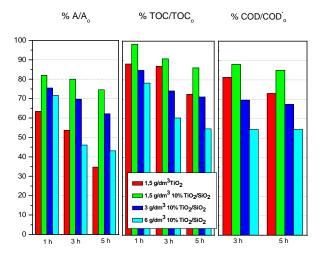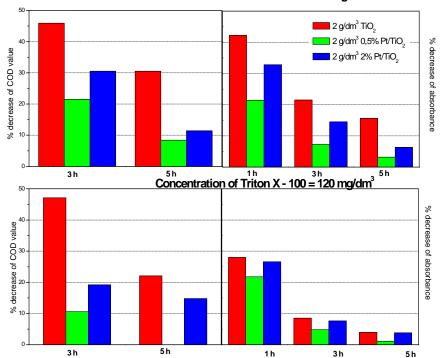

Fig. 5. The influence of photodecomposition parameters of Triton X-100 water solution expressed as: A/A_0, TOC/TOC_0, COD/COD_0 in function of reaction time at different doses of 10% TiO_2/SiO_2 catalyst (1.5, 3 and 6 mg/dm³). The measurements were carried out using 150 W lamp and the initial surfactant concentration of 120 mg/dm³

The influence of the reaction time on Triton X-100 decomposition (A/A_0, TOC/TOC$_0$ and COD/COD$_0$) at different doses of 10% TiO$_2$/SiO$_2$ catalyst (1.5, 3 and 6 mg/dm^3) is presented in figure 5. The measurements were carried out using 120 W lamp and an initial surfactant concentration of 120 mg/dm^3.

The modified platinum-supported catalyst Pt/TiO$_2$ was powdered in mortar before its use as photocatalyst for two initial concentrations of Triton X-100 solutions (120 mg/dm^3 and 250 mg/dm^3) and the examples of the results obtained are presented in figure 6.

Fig. 6. The absorbance ratio A/A_0 and chemical oxygen demand ratio COD/COD$_0$ for two Triton X-100 water solutions (120 and 250 mg/dm^3) expressed as: A/A_0 TOC/TOC$_0$, COD/COD$_0$ in function of reaction time at 2 mg/dm^3 dose of photocatalysts: TiO$_2$, 0.5% Pt/TiO$_2$ and 2% Pt/TiO$_2$. The measurements were carried out using 150 W lamp

Titania–silica (10% TiO$_2$/SiO$_2$) and platinum-supported (0.5% and 2% Pt/10% TiO$_2$/SiO$_2$) catalysts were tested in Triton X-100 solution decomposition. The tests were carried out at the initial Triton X-100 concentration of 120 mg/dm^3 and UV lamp of 150 W. The results obtained, i.e., the absorbance ratio (A/A_0) and chemical oxygen demand ratio COD/COD$_0$, are given in figure 7.
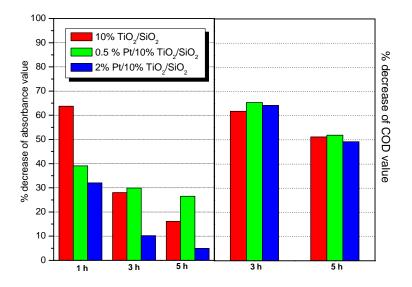
Fig. 7. The absorbance ratio A/A_0 and chemical oxygen demand ratio COD/COD_0 for titania–silica
(10% TiO_2/SiO_2) and platinum-supported (0.5 and 2% Pt/10% TiO_2/SiO_2) catalysts.
The measurements were carried out using: 150 W lamp, the photocatalyst dose of 1.5 g/dm^3
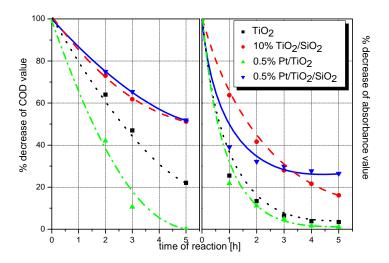and an initial surfactant concentration of 120 mg/dm^3

Fig. 8. The kinetic curves of the photocatalysts applied: TiO_2, 0.5% Pt/TiO_2, 10% TiO_2/SiO_2
and 0.5% Pt/10% TiO_2/SiO_2 during oxidation of Triton X-100 aqueous solution. An initial surfactant
concentration was 120 mg/dm^3. The dose of photocatalyst was 2 g/dm^3 and UV lamp of 150 W was used

The kinetic behaviour of such photocatalysts as TiO_2, 0.5% Pt/TiO_2, 10%
TiO_2/SiO_2 and 0.5% Pt/TiO_2/SiO_2 is presented in figure 8. The initial surfactant con-

centration was 120 mg/dm^3, the dose of photocatalyst reached 2 g/dm^3 and UV lamp of 150 W was used.

During the Triton decomposition the surface of photocatalysts did not change at all.

4. DISCUSSION

Photocatalytic oxidation of the aqueous solution of Triton X-100 was the subject of our previous study [21]. One of its disadvantages is a periodic character of photocatalyst suspension requiring titania separation after reaction completion. In order to resolve this problem, TiO_2 was dispersed on the silica support and the accessible surface of 50 m^2/g was increased up to 380 m^2/g. The initial material, i.e. silica, for 10% TiO_2/SiO_2 catalyst has grains in the range of 1–3 mm, but during TiO_2 deposition the catalyst grains stick together keeping a total surface area constant. This kind of catalyst makes separation fast and easy.

The experiments in Triton X-100 decomposition confirmed high catalytic activity of this catalyst due to its granulation. The optimal catalyst dose was 1.5 g of TiO_2 per one dm^3 of solution [21]. In the case of 10% TiO_2/SiO_2, the changes of the absorbance (A), chemical oxygen demand (COD) and total organic carbon (TOC) lead to the conclusion that pure TiO_2 faster decomposes Triton X-100 than the same dose of TiO_2/SiO_2 catalyst. The differences in the absorbance, COD and TOC values range from 12% after one hour up to 37% after 5 hours of reaction (see figure 5). Better results are obtained at high loading of photocatalyst. At the catalyst 10% TiO_2/SiO_2 dose of 3 g/dm^3 higher values of COD and TOC reduction are obtained, whereas at its dose of 6 g/dm^3 the differences approach 20% after 5 hours of reaction. The changes of absorbance $\lambda = 224$ nm, being cumulative measure of Triton X-100 and unsaturated and aromatic compounds, are similar for both TiO_2 and 10% TiO_2/SiO_2 catalysts. The preparation of 10% TiO_2/SiO_2 catalyst is easier because surfactant molecules undergo deep decomposition on interior surface of silica pores before the final products are desorbed to liquid phase. Different pathway is postulated on the surface of TiO_2 catalyst, where after primary adsorption–oxidation stage a desorption takes place and in turn successive adsorption of subsequent surfactant molecules follows.

In order to improve photocatalyst activity, both TiO_2 and 10% TiO_2/SiO_2 catalysts were additionally platinum-promoted (0.5 and 2 wt %). Platinum-modified titania does not change practically its surface area of about 49 m^2/g in comparison with that characteristic of TiO_2 alone, i.e., 50 m^2/g (see the table). A decrease more than 10% in surface area is observed when SiO_2 itself is compared with silica covered with titania (10% TiO_2/SiO_2) which is a result of partial blocking of silica pores by titania. The diameter of pores that predominate decreases from 50 nm for SiO_2 to about 40 nm for 10% TiO_2/SiO_2. The 10% TiO_2/SiO_2 photocatalysts obtained have much

higher bulk density and even after mechanical powdering the density was three times higher than that characteristic of pure titania.

Pt/TiO$_2$ catalysts of lower metal loading (0.5% Pt) appeared to be more active than catalyst containing 2% Pt (figure 6). After 5-hour oxidation of Triton X-100 in an initial concentration of 120 mg/dm^3 in the presence of 0.5% Pt/TiO$_2$ catalyst the reduction of COD value was 100%, whereas 85% reduction was measured for 2% Pt/TiO$_2$ and 79% for TiO$_2$ alone. The same dependence is observed for more concentrated solution of the surfactant (250 mg/dm^3) which seems to confirm that micellar form of Triton X-100 does not influence its decomposition. After 5 hours of reaction almost 90% reduction of COD was observed due to UV radiation of more powerful 150 W lamp.

In the case of platinum supported on titania/silica, higher activity was found for 2% Pt/10% TiO$_2$/SiO$_2$ catalyst, especially in an initial period of reaction. One can suppose that destruction of aromatic ring is equivalent to Triton molecule decomposition, but total oxidation of the solution take place with similar efficiency.

The efficiency of decomposing the Triton X-100 solution (120 mg/dm^3) in the presence of four different photocatalysts: TiO$_2$, 0.5% Pt/TiO$_2$, 10% TiO$_2$/SiO$_2$ and 0.5% Pt/10% TiO$_2$/SiO$_2$ is compared in figure 8. TiO$_2$/SiO$_2$ and 0.5% Pt/TiO$_2$/SiO$_2$ catalysts reduced COD in a comparable amount, which reached 50% after 5 hours. TiO$_2$ catalyst appeared to be more efficient, whereas 0.5% Pt/TiO$_2$ catalyst was most active, resulting in a COD value close to zero after 5 hours. The same, even more profound, tendency was observed at A/A_0 ratio. After 2 hours of reaction the solution mineralization blocking was found for 0.5% Pt/10% TiO$_2$/SiO$_2$ catalyst.

Summing up, the platinum-supported titania catalysts appeared to be active in decomposing aqueous solution of Triton X-100. Even more interesting are Pt/TiO$_2$ catalysts supported on large surface area of silica carrier. About a twofold increase in the catalyst activity can be attained, but the amount of TiO$_2$ is 10 times lower than in the case of pure titania catalyst. Higher catalyst loading can increase the efficiency of catalytic system. A proper choice of the catalyst that achieves its optimum activity requires further comparative study of its efficient mechanistic properties.

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FOTOKATALITYCZNY ROZKŁAD TRITONU X-100
W ROZTWORZE WODNYM Z UDZIAŁEM TIO$_2$ AKTYWOWANYM PLATYNĄ

Aby zwiększyć efektywność działania dwutlenku tytanu jako fotokatalizatora, poddaje się go aktywacji za pomocą takich metali szlachetnych jak platyna i pallad. Stosowanie metali jako promotorów katalizatorów w celu zwiększenia ich efektywności jest znanym i od dawna stosowanym zabiegiem. Nanoszenie zarówno platyny w ilości 0,5% i 2% wagowych na TiO$_2$ (P25 firmy Degussa), jak i TiO$_2$ na krzemionkę pozwoliło zwiększyć rozkład Tritonów, tj. niejonowych środków powierzchniowych. Gdy jako katalizator zastosowano TiO$_2$ z naniesioną warstwą platyny w ilości 0,5% wagowego, zwiększono redukcję wartości ChZT o około 20% w porównaniu z wynikami uzyskanymi dla czystego TiO$_2$. Naniesienie TiO$_2$ na krzemionkę ułatwiło proces separacji katalizatora po reakcji, a dotowanie go platyną spowodowało efektywny rozkład związków powierzchniowo czynnych.