

Comparison of Different Characteristics of TiO₂ Films and Their Photocatalytic Properties[†]

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Abstract

TiO₂ is known as a photocatalyst and is often utilized for the oxidative mineralisation of pollutants. The correlation between the thickness, mass, surface topography and the type of support of the titania films and their photocatalytic activities were studied in this work. TiO₂ films were deposited on two different glass supports via sol-gel methods. Film thicknesses and weights have been measured. Film surfaces were characterized by AFM measurements. The presence of surfactant in the sol and the type of support influence the morphology of film's surface and the UV absorption characteristics. The addition of surfactants also increases the thicknesses of calcined films. For the photocatalytic experiments the titania film was immersed in an aqueous solution of Plasmocorinth B. Sodium glass support reduces the photocatalytic activity compared to protected glass support with a thin silica layer. The photocatalytic activity was found to increase with the number of TiO₂ layers in all cases. However, when comparing different films with the same number of TiO₂ layers, the addition of Pluronic F-127 surfactant in the sol was shown to have a significant positive effect on the photocatalytic activity. The use of this surfactant results in thicker and heavier films and consequently in more efficient absorption of UVA light.

Key words: TiO₂ thin films, UV-Vis, surface morphology, photocatalysis, azo dye.

Introduction

Conventional water purification methods often do not successfully deal with chemically and microbiologically stable synthetic organic compounds that are dissolved in water at low concentrations (textile dyes, agricultural or industrial effluents). In order to cleanse water of these extra-stable organics, so-called Advanced Oxidation Methods (AOMs) have been developed. Most AOMs are ambient-temperature processes that use energy to produce highly reactive intermediates of high oxidizing or reducing potential, which then attack and destroy the target compounds.

TiO₂ photocatalysis belongs to AOMs. The photocatalytic behaviour of TiO₂ has been the subject of intensive studies for more than 20 years.¹⁻³ Basically, the photocatalytic process is initiated by the photogeneration of hole/electron pairs in the semiconductor by absorption of UV-light with energy equal to or higher than the corresponding band gap. Electrons and holes can either recombine, dissipate energy, or be available for redox reactions with electron-donor or acceptor species, adsorbed at the semiconductor surface or localized in the electrical

double layer surrounding the particle. In the second scenario, positive holes oxidize water molecules or OH⁻ ions with the formation of highly reactive hydroxyl radicals. On the other hand adsorbed oxygen molecules react with negative electrons producing superoxide anions, which can be in the following steps converted to hydrogen peroxide and also to hydroxyl radicals, both able to oxidize stable organic molecules.

TiO₂ is mostly used in a powder form for photocatalytic experiments.^{4,6} However, the major drawback of slurry-type photoreactors is the final particle-fluid separation for the catalyst recycling. If TiO₂ is immobilized on a rigid support as a thin (nano- or micro-scale) film, such a problem is avoided.

A large number of studies have been made in the last ten years to immobilize the catalyst on a rigid support via different deposition techniques, such as chemical or physical vapour deposition, spray pyrolysis, sol-gel technique etc.³ Sol gel process is among all other processes most frequently used because of its relatively low cost and flexible applicability to wide range of size and shape of substrate (deposition on optical fibers⁷, spherical ceramic balls⁸, glass rods⁹ etc). However, there are certain problems regarding

Table 1. Composition of titania sols

the symbol of titania sol	Ti precursor - Ti(OiPr) ₄	complexing agent	solvent - MeOCH ₂ CH ₂ OH	surfactant
U	8.96 mL	AcOH 1.74 mL	30.4 mL	/
R	8.96 mL	AcOH 1.74 mL	30.4 mL	Brij 56 7 g
D	8.96 mL	EAA 3.94 mL	32.4 mL	/
C	8.96 mL	EAA 3.94 mL	32.4 mL	Pluronic F-127 4.7 g

the photocatalytic activity of the immobilized TiO₂ i.e. (i) a lower rate of electron and hole transfer across the semiconductor-solution interface because of the decreased catalyst's surface in immobilized TiO₂; (ii) light absorption characteristics because of the limited amount of TiO₂ particles capable of absorbing the light; (iii) a substrate since it may have a negative influence on the photocatalytic activities of the titania films.³

Nanostructured materials are preferred over dense structures, because of their larger active surface. The production of nanostructured films is nowadays an established method and TiO₂ nanoparticles and mesoporous films are among the materials routinely produced through the sol-gel chemistry processing.¹⁰⁻¹⁴ It is very important to prepare TiO₂ films that are capable of harvesting incident light to a great extent, and at the same time films with a high surface area and porosity to increase the rate of reaction between photogenerated species and the pollutants.¹⁵⁻¹⁸

In this work, we have tested the photocatalytic activities of TiO₂ films prepared by dip-coating deposition from sols containing two different surfactants as templating agents. The effects of different supports, thickness, weight and surface structure of the films on their efficiencies for photocatalytic decolorisation of an aqueous solution of azo dye were evaluated. Plasmocorinth B, a member of a group of azo dyes was chosen as a model compound. It is known that toxicity and potentially carcinogenic nature of azo dyes and their precursors represent an increasing danger to aquatic life. Therefore the investigations of azo dye degradation are important. Several studies still nowadays deal with the photocatalytic treatment of aqueous wastes containing single or mixed azo dyes.¹⁹⁻²²

Experimental

1 Preparation of sols

SiO₂ sol: Tetraethoxysilane (TEOS) (15.0 g) was dissolved in ethanol (EtOH) (22.0 g). Separately 11.5% solution of nitric(V) acid was prepared by dissolving concentrated (65%) nitric(V) acid (1.85 g) in water (10.5 g). After ten minutes both solutions were mixed together and the resultant sol was used after ageing at room temperature for two hours. The sol was unstable,

it gelled in one day and therefore it was always freshly prepared.

TiO₂ sols: Titanium(IV) isopropoxide (Ti(OiPr)₄) was added to acetic acid (AcOH) or to ethyl acetoacetate (EAA) during constant stirring. The prepared solution was dissolved in 2-methoxyethanol (MeOCH₂CH₂OH) after 5 minutes.²³ The resultant alkoxide solution was stirred at room temperature for solvolysis and condensation reactions at least for three hours. After using, the sol was kept in the refrigerator. It was conditioned at room temperature for at least one hour before the next use.

The procedure for the preparation of the sols with added surfactants was basically the same as described above. The only difference was that the surfactant, either polyoxyethylene(10) cetyl ether (Brij 56) or triblock copolymer Pluronic F-127, was dissolved in the sol after the addition of MeOCH₂CH₂OH.²⁴

The quantities of particular chemicals, used for different TiO₂ sols preparation, are given in Table 1.

2 Deposition of films

TiO₂ films: Soda-lime glass plates (75 mm x 25 mm x 1 mm) were washed with ethanol and dried before use. The TiO₂ films were deposited on them using the dip-coating technique with a withdrawal speed of 10 cm/min. The substrates coated with gel films were left at room temperature in air for 30 min and then they were calcined at 500 °C for 30 min. The thickness of the TiO₂ films was increased by repeating the dipping and heat-treatment cycle up to five times.

SiO₂ protecting layer: The SiO₂ protective layer was deposited on the sodium glass support from SiO₂ sol by the dip-coating technique in an ambient atmosphere. The withdrawal speed was 5 cm/min. The substrates coated with gel films were left at room temperature in air for 30 min and then they were calcined at 500 °C for 30 min. TiO₂ films were deposited on SiO₂ protective layer as it is described above.

3 Characterization of films

Thicknesses of TiO₂ films deposited from sols U, R, D and C on SiO₂ precoated sodium glass by 1, 3 and 5 coating-heating cycles have been measured. For this purpose, a part of the coating was removed from the

substrate by a mixture of zinc powder and concentrated HCl until the sharp step was obtained between the substrate and the intact TiO₂ film. Thicknesses of titania films were measured with a Talysurf profilometer (Taylor – Hobson). Absorption spectra (340 - 400 nm spectral range) of clean and calcined both-sided TiO₂ films deposited on two different supports were recorded on HP 8453 UV-Vis spectrophotometer. Sodium glass, protected with SiO₂ layer, was used as a blank. The band gap energies were calculated and estimated from UV-transmission spectra of the titania films according to Sreemany and Sen.²⁵

Surface topography of TiO₂ films made by 1 dipping-heating cycle was evaluated by atomic force microscopy (AFM) on CP-II scanning probe microscope (Veeco) in non-contact mode at constant force in air. The tip was also a Veeco product (model RFESPA-M).

4 Photocatalytic experiments

A flow cell was constructed from Teflon and borosilicate glass with the capability of purging with different gases, cooling with tap water and on-line absorbance measuring of the solutions. A 335 nm cut-off filter was used in front of the photocatalytic cell. A 125 W Xe lamp (Cermox xenon parabolic lamp) was used as a light source. One side of TiO₂ thin film (23 mm x 23 mm) was irradiated along the normal direction. Aqueous solution (4.8 mL) of Plasmocorinth B – 3-(5-chloro-2-hydroxyphenylazo)-4,5-dihydroxy-2,7-naphthalene disulfonic acid, disodium salt (insert in Figure 5) – (40 mg/L) was used as a photodegradation medium in the experiments. The solution was constantly purged with oxygen during the irradiation. The peristaltic pump Heidolph PD 5001 with silicon hose was used to drive the solution with the rate of 10 mL/min from the flow cell to the sample compartment of the UV-Vis spectrophotometer and back to the flow cell. Thus, on-line measuring of the absorbance of the solution was possible.

Results and discussion

1 Film characterization

It is expected that more viscous sol is obtained when surfactant with higher molecular weight is added to the primary sol. Pluronic F-127 has molecular weight of 12600 g/mol and it is a much larger molecule than, for example, Brij 56 with molecular weight of 363 g/mol. The expectations were confirmed, since thicker and crack-free films were obtained from sols with added Pluronic F-127. The other observations were that linear dependence exists between the thicknesses of the films and number of layers (Figure 1) and that the thicknesses of the films, deposited from the same sol on different supports, are the same.

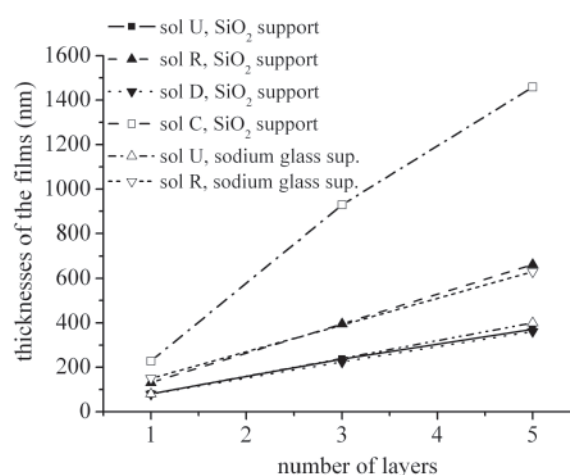


Figure 1. The influence of number of TiO₂ layers on thicknesses of the titania films

Along with a thickness increase, the mass of titania films also increased when using surfactants (Table 2). An interesting observation was that the density of the film, deposited from the sol with added Pluronic F-127, was only half as dense as film without added Pluronic F-127. Supposing that we had the same crystalline phase in both cases (anatase) it could be concluded that the film with added Pluronic F-127 in the sol was more porous than the others. The presence of anatase phase in all cases was confirmed with XRD measurements of titania films.²⁴ Another contribution to lower density of the films obtained by using surfactants may be also the higher portion of amorphous phase (having undefined structure and density) in the templated films, where the crystallization process is retarded with regard to the crystallization in films made without surfactants.²⁴

Table 2. The measured masses, calculated surface densities and densities of four-layered titania films, deposited on SiO₂ support

sol	titania mass (mg)	surface density (g/m ²)	density (g/dm ³)
U	3.0	1.4	4200
R	3.6	1.8	3300
D	3.0	1.3	4300
C	5.8	2.6	2200

To observe the differences in film morphologies, AFM images were taken (Figure 2). The addition of surfactant into titania sols increases the thickness of the films (Figure 1) and also selectively influences their surface roughness. The films, deposited from sol C, have twice as high surface roughness as the films, deposited from sols U or D.

Results revealed also that the surface morphology of the films depends on the substrate (Figure 2, Table 3). A different morphology noted for the films deposited on glass support without the SiO₂ protecting layer (Figure 2 A), indicated the influence of the glass support. This effect can be attributed to the diffusion of Na⁺ ions from bare glass support in the interior of the film contributing to the formation of a mixed oxide phase²⁶ and larger anatase grains.^{24,27} The effect is the most pronounced for films deposited from sol R and less for films deposited from sol U or D. It seems that there is a simultaneous effect of sodium diffusion and the surfactant on growth of the nano grains of TiO₂.

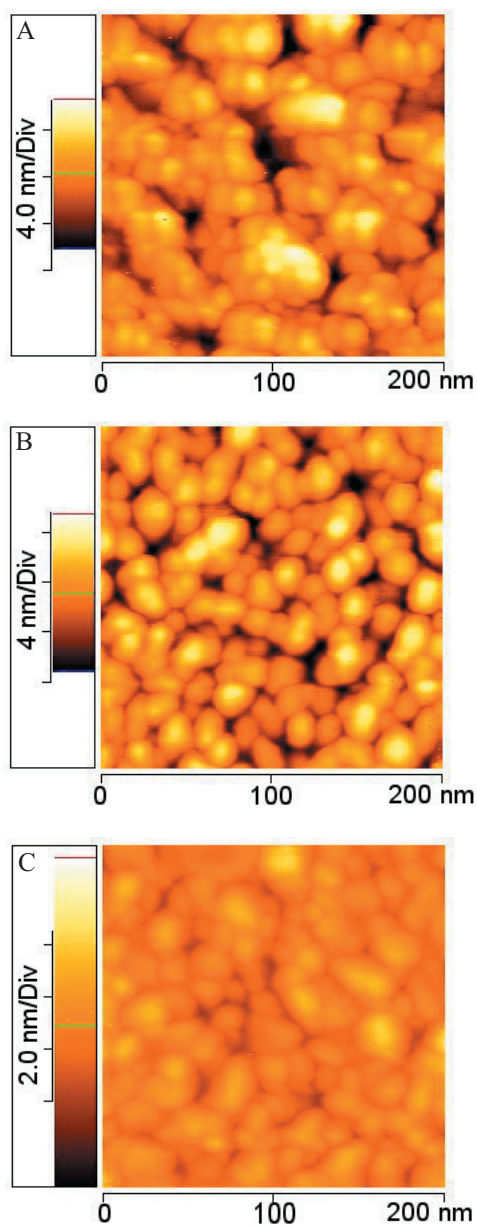


Figure 2. AFM images (200 nm x 200 nm) of calcined, single-layered TiO₂ films: A) sol C, sodium glass support; B) sol C, SiO₂ support; C) sol D, SiO₂ support

Table 3. Thicknesses and surface roughnesses (calculated from AFM images) of single-layered titania films

<i>sol and support</i>	<i>thickness (nm)</i>	<i>RMS roughness (nm)</i>
U, SiO ₂ support	80	1.23
U, sodium glass support	80	0.90
R, SiO ₂ support	131	1.29
R, sodium glass support	150	3.62
D, SiO ₂ support	79	0.95
D, sodium glass support	/	0.85
C, SiO ₂ support	228	1.87
C, sodium glass support	/	2.21

Similarly, light absorption properties of titania films depend not only on the sol, used for preparation of the films, but also on the type of the substrate. Films deposited on sodium glass support obtained by the single deposition cycle absorb much less light between 300 and 400 nm than the same films, deposited on SiO₂ support irrespectively of the addition of surfactant. UV spectra of films deposited from sol R are represented in Figure 3. Interestingly, the difference of the absorption becomes much lower when four-layered films are studied.

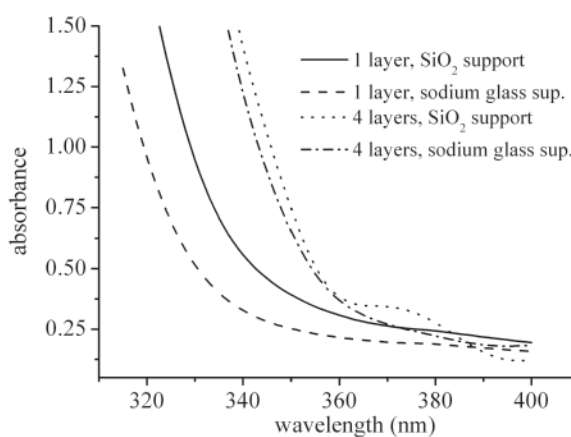


Figure 3. The dependence of the support used for the preparation of TiO₂ films on their UV absorption characteristics; sol R

A similar set of experiments was performed also with different films deposited on SiO₂ support. The absorption of light increases with the number of layers of TiO₂ films regardless the use of surfactant. Figure 4 shows the results obtained with films deposited from sol with added surfactant Pluronic F-127. An increase in the number of dippings produces a red-shift of the absorption edge of the TiO₂ film which involves a decrease of the band gap energies from 3.49 eV (1 layer) to 3.31 eV (5 layers). On the other hand, when comparing the films with the same number of layers the

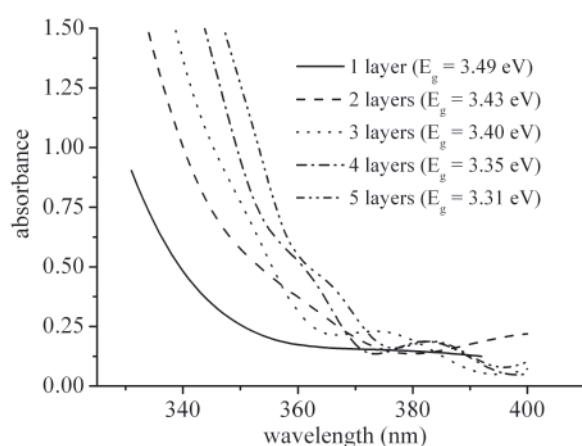


Figure 4. The dependence of number of layers of TiO_2 on UV absorption properties of TiO_2 films with added surfactant Pluronic F-127 in the sol (sol C); SiO_2 support

addition of Pluronic F-127 results in a higher absorption of light between 300 and 400 nm. This is true for films made by 1, 2, 3, 4 or 5 dipping cycles.

2 Photocatalytic experiments

Plasmocorinth B was used as a model azo compound in photocatalytic experiments. Figure 5 represents the degradation curves of the dye solution catalyzed by one-layered TiO_2 films deposited on SiO_2 support. The same experiments were done also with two-, three-, four- and five- layered films, deposited on SiO_2 support, and with one- and four-layered films, deposited on sodium glass support. All degradation curves indicate first – order kinetics, therefore they were fitted as first order reactions regarding the decrease of absorbance at 527 nm. The mean half-times and standard deviations were calculated for each catalyst separately. Some of the results are included in Table 4.

Temperature of calcination of the films was 500 °C, which is the optimal temperature for sodium glass substrate. Lower temperatures of annealing result in lower photocatalytic activity of photocatalyst since the photocatalytically active TiO_2 crystalline phase (anatase) is not yet developed in the films at lower annealing temperatures. Higher temperature would result in melting of sodium glass support. Time of calcination was 30 minutes, which was long enough to degrade all the organic residues captured in the film. The highest photodegradation rates were achieved when the solution was purged with oxygen during the experiment. Purging with argon or no purging resulted in lower degradation rates.

We compared the films deposited from the same sol on both substrates. Na^+ cations from the glass substrate diffuse into the TiO_2 film's grains during the thermal treatment with an adverse effect on the photocatalytic

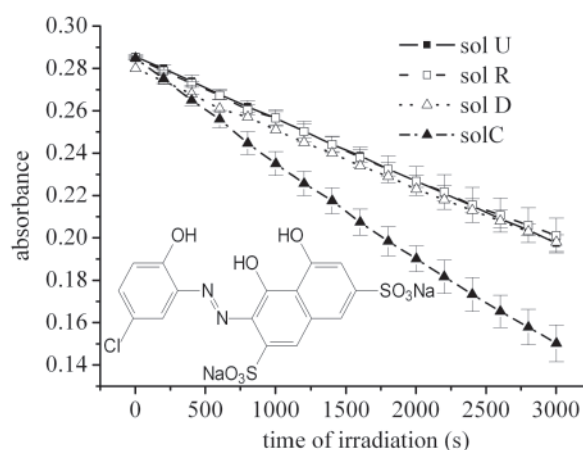


Figure 5. The influence of addition of surfactants in sols on photodecolorisation of Plasmocorinth B solution with one-layered TiO_2 films deposited on SiO_2 support; insert: chemical structure of Plasmocorinth B

activity.³ This was truly noticed also in our experiments. The only photocatalytically active single-layered titania film was the one deposited from sol with added Pluronic F-127. Its photocatalytic activity was also lower than the activity of the same film, deposited on SiO_2 support (lines 7 and 8 in Table 4). Similar observations were noticed also with four-layered films. The films deposited from sols D and U (lines 10 and 14 in Table 4) were still not photocatalytically active. The other two films (lines 12 and 16 in Table 4) showed some activity against the photodecolorisation of Plasmocorinth B, but this again was still lower than with the same films, deposited on SiO_2 support.

Table 4. The calculated half-times and rate constants of photodecolorisation of aqueous Plasmocorinth B solutions using calcined TiO_2 films (1 and 4 layers)

	<i>sol</i>	<i>number of layers</i>	<i>support</i>	<i>half-time - mean value (s)</i>	<i>rate constant ($\text{s}^{-1} \times 10^{-4}$)</i>
1	U	1	SiO_2	6500	1.07
2	U	1	sodium glass	∞	0
3	R	1	SiO_2	6500	1.07
4	R	1	sodium glass	∞	0
5	D	1	SiO_2	6500	1.07
6	D	1	sodium glass	∞	0
7	C	1	SiO_2	3700	1.87
8	C	1	sodium glass	6400	1.08
9	U	4	SiO_2	2200	3.15
10	U	4	sodium glass	∞	0
11	R	4	SiO_2	1750	3.96
12	R	4	sodium glass	2250	3.08
13	D	4	SiO_2	2150	3.22
14	D	4	sodium glass	∞	0
15	C	4	SiO_2	1150	6.03
16	C	4	sodium glass	1300	5.33

By increasing the thickness of the deposited films obtained by performing multiple dipping cycles, the effect of sodium diffusion diminishes and the upper part of the TiO₂ film becomes more or less free of sodium ions impurities and the films become photocatalytically active.

Accordingly, a set of experiments was performed with different films deposited on SiO₂ support. Results presented in Table 4 show that the photocatalytic activity of the films when deposited on the glass slides with the SiO₂ protective layer increases with the film thickness (Figure 1) in all cases. On the other hand it is shown that when comparing different films with the same number of layers of TiO₂, the addition of Pluronic F-127 has a noticeable effect on the increasing photocatalytic activity of the films when deposited on the glass slides with the SiO₂ barrier film.

Our photodegradation studies showed that photocatalytic activity of the films increases with the film thickness and consequently with the film's mass irrespective of addition of surfactants into the sols (Figure 6). The relation between the thickness (the number of layers, the mass of titania film) and the degradation rate is quite linear for films, deposited from sols U, R and D. This could be simply accounted for by the increase in the number of active sites and in the amount of photons absorbed by TiO₂. Another proof for this are UV-Vis spectra of the films (Figure 4). Comparison of film thicknesses and the UV-Vis spectra shows that thicker layer of TiO₂ results in increased absorbance of the film in UV region.

On the contrary, the rate constants of films, deposited from sol C, do not increase linearly with the number of coatings in the whole range (Figure 6). Films from sol C are thicker than films deposited from sols U and D and photogenerated holes and electrons from the bottom of the films could not reach the surface of the films, where they can react with the organic molecule.

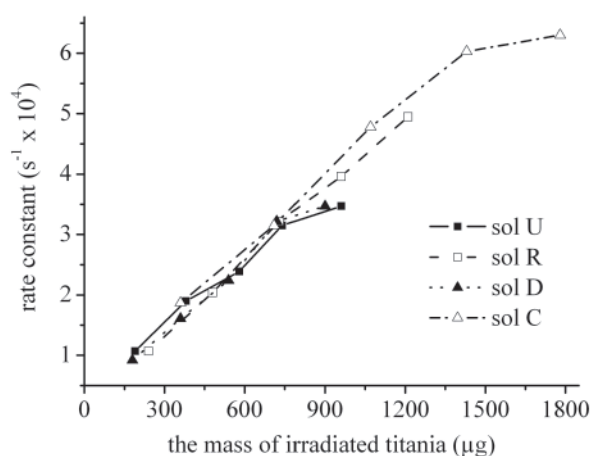


Figure 6. The influence of irradiated titania mass on the rate constant for the photodecolorisation of aqueous solution of Plasmocorinth B; SiO₂ support

Therefore the photocatalytic activity cannot correlate infinitely with the film thickness.

The different masses of irradiated titania films are involved also when comparing different TiO₂ films with the same number of layers and their photodegradation rates. It was calculated from degradation rates that one-layered TiO₂ film deposited from sol C (surfactant Pluronic F-127) is twice more photocatalytically active than similar film deposited from sol D (without surfactant). The mass of the first film is 360 µg, the mass of the second is 180 µg. Higher mass means more photoactive sites and more efficient absorption of light and consequently higher number of reactive species on the surface of the film capable of reacting with azo dye. It seems from the Figure 6 that the mass of titania is the only parameter defining the final photocatalytic activity, because all deposited films have similar photocatalytic activity per unit of mass. This was not expected because it was believed that surface structure and the porosity of the film could also be involved in improving the photocatalytic activity. More work is planned in investigations of the influence of porosity on photocatalytic activities of the films (e.g. measuring the photocatalytic activities of well-ordered mesoporous titania films).

Conclusions

Results reveal that the surface morphology of the films depends on the surfactant used and on the substrate. Adding surfactant in the sol increases dip coating efficiency and presumably also the porosity of prepared films. Increased thickness (mass) of titania films means more efficient absorption of light and consequently higher number of reactive species of the film capable of reacting with azo dye. Additional layers of TiO₂ also result in higher thickness of the films and consequently in higher photocatalytic activities of the prepared films but the photodegradation rates do not increase linearly with the number of coatings, because photogenerated holes and electrons from the bottom of the thicker films recombine before they reach the surface of the films, where they can react with the organic molecule. For the films made with surfactant the increase is linear in the broader thickness range.

Sodium glass support results in a decrease of photocatalytic activities of the films because of the diffusion of sodium cations into the titania layer during the heat treatment. The negative influence of sodium cations could be observed in UV spectra of prepared films, where those films deposited on sodium glass support absorb much less UV light than the films deposited on SiO₂ support. Films deposited on sodium glass show some photocatalytic activity only if they are thick enough.

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Povzetek

TiO₂ je znan fotokatalizator, ki ga pogosto uporabljamo pri oksidativni mineralizaciji organskih onesnaževal. Namen naše raziskave je bila primerjava med različnimi lastnostmi pripravljenih TiO₂ filmov in njihovimi fotokatalitskimi aktivnostmi. TiO₂ filme smo nanašali na dve različni podlagi s pomočjo sol-gel tehnike. Merili smo debeline in mase tako pripravljenih žganih filmov. Površine filmov smo okarakterizirali z mikroskopom na atomsko silo. Uporaba surfaktanta in vrsta podlage vplivata na morfologijo in na UV absorpcijske lastnosti filmov. Dodatek surfaktanta v sol vpliva tudi na povečano debelino žganih filmov. V tipičnem fotokatalitskem eksperimentu smo obsevali TiO₂ film v prisotnosti vodne raztopine Plazmokerinta B. Natrijevo steklo kot substrat zmanjša fotokatalitsko aktivnost filmov v primerjavi s SiO₂. Fotokatalitska aktivnost vseh filmov raste s številom plasti TiO₂, ko pa smo primerjali različne filme z enakim številom plasti, smo ugotovili, da dodatek Pluronic F-127 v solu pozitivno vpliva na fotokatalitsko aktivnost. Z uporabo surfaktanta dobimo debelejšje in težje filme, kar posledično pomeni večjo učinkovitost absorpcije UVA svetlobe.