

Doctoral (Ph.D.) Theses

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Photoactivity of surface-functionalized nanocrystalline  
titanium-dioxide at solid/gas and solid/liquid interfaces

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## **Introduction and Objectives**

Accumulation of environmentally harmful and not readily biodegradable compounds both in surface waters and in the atmosphere prompts the development of efficient water and air purification technologies. Heterogeneous photocatalysis has already proven to be efficient in the degradation of numerous organic substrates. Regarding practical applicability, the ideal semiconductor metal oxide photocatalyst is chemically stable, easy to prepare and use, safe for human and environment, efficiently catalyses the reaction and it is cheap to produce. Among various metal oxides ( $\text{TiO}_2$ ,  $\text{ZnO}$ ,  $\text{WO}_3$ ,  $\text{SnO}_2$  etc.) titanium dioxide meets nearly all of the above requirements. Therefore, study of this semiconductor was the straightforward choice as the subject of my dissertation.

Many research groups have focused on recently the improvement the photocatalytic activity of nanostructured titanium dioxide and on its excitation by visible light. Doping and surface modification of  $\text{TiO}_2$  with this aim, using a variety of elements, has become one of the most intensively studied research areas. The application of non-metals for this purpose has been recent but has already yielded many remarkable results. Based on the promising predictions of the pertinent literature as well as on my preliminary results, my dissertation focused on the phosphate modification of titanium dioxide.

The main objective of my experimental work was to prepare photoactive titanium dioxide samples by using colloid chemical techniques, such as the sol-gel method and to explore the relationship between the structural and photocatalytic properties of the oxides obtained under systematically varied experimental conditions (phosphate content, calcination temperature).

The effect of surface modification on the particle size, specific surface area, porosity, crystalline variety, thermal stability, morphology and photoconductivity of the titanium dioxide were studied using the available instrumental techniques. Photoactivities of titanium dioxide samples were compared at 25 °C at the solid/liquid (S/L) interface in the photooxidation reaction of phenol and at the solid/gas (S/G) interface in ethanol photooxidation. For studying the gas phase oxidation of organic substrates, a photoreactor that allows easy and reliable comparison of the activities of various catalysts were developed and designed.

## Materials and methods

Phosphate-modified titanium dioxide (P-TiO<sub>2</sub>) samples of varying phosphate contents were prepared by sol-gel method. The titanium(IV) isopropoxide was hydrolysed and the amorphous TiO<sub>2</sub>·nH<sub>2</sub>O obtained was treated with phosphoric acid solutions. The titania powders were calcined at the range of 100-900 °C. The chemisorbed phosphate species on the surface of the P-TiO<sub>2</sub> samples was removed by a treatment of 1M NaHCO<sub>3</sub> solution at 60 °C for 24 h.

The Ti and P content of the samples were determined in an all-argon sequential Jobin-Yvon 24 (France) inductively coupled plasma atomic emission (ICP-AES) spectrometer, equipped with a Babington V-groove Teflon nebulizer and a Miniplus III (Gilson, France) peristaltic pump. The intensity of the spectral lines of 213.62 (P I) and 337.28 (Ti II) nm were measured.

Thermogravimetric investigations carried out in air with a TGA/SDTA 851° (Mettler Toledo) derivatograph applying a heating rate of 5 °C min<sup>-1</sup>.

X-ray diffraction (XRD) patterns were collected on a Philips PW 1830 powder diffractometer using CuK $\alpha$ -radiation ( $\lambda=0.1542$  nm).

The SAXS measurements of powders were carried out in vacuum by a compact Kratky camera (KCEC/3) with a position sensitive detector (Mbraun, PDS 50M).

The study of porosity and surface area was performed on a Gemini 2375 (Micromeritics) instrument by recording N<sub>2</sub>-sorption isotherms at 77 K. Specific surface areas were calculated using the BET (Brunauer–Emmett–Teller) model.

Transmission electron microscopy (TEM) images were taken using a Philips CM-10 electron microscope, using an accelerating voltage of 100 kV.

Morphology of the titania samples was studied by a Hitachi, S-4700 scanning electron microscope. The titanium and phosphorous contents of the samples were characterized by an RönTec QX2 energy dispersive spectrometer (EDS).

UV-vis-DR spectra were recorded in a Unicam 8700 spectrometer using a Thermo-Electron RSA-UC 40 diffuse reflectance cell. The samples were mixed with dry KBr (2 wt %), and pure dry KBr was used as reference.

XP spectra were taken with a SPECS instrument equipped with a PHOIBOS 150 MCD 9 hemispherical electron energy analyzer operated in the FAT mode.

DRIFT measurements were performed in a Bio-Rad Digilab Division FTS-65A/896 spectrometer, taking 256 scans per sample with  $4\text{ cm}^{-1}$  resolution. The samples were mixed with dry KBr (2 wt %), and pure dry KBr was used as reference.

The photoconductivity of titania films was measured on interdigitated electrodes (Curamik, Germany) with 140 mm meander length. A 200 W xenon-mercury lamp (Hamamatsu, Lightingcure LC4 L8444-01) was used as a light source ( $\lambda_{\text{max}}=365\text{ nm}$ ). The photocurrent response during illumination was measured by a Keithley 2400 Source Meter recorded every 1s by a computer via RS232 port.

Photooxidation of ethanol at solid-gas interface was performed in a reactor with volume  $\sim 700\text{ ml}$  at  $25 \pm 0.1\text{ }^{\circ}\text{C}$ . The light source of the reactor was a 15 W low-pressure mercury lamp (GCL307T5L/CELL LightTech, Hungary) with characteristic emission wavelength at  $\lambda_{\text{max}} = 254\text{ nm}$ .

The composition of the gas phase was analyzed at given time intervals in a gas chromatograph (Shimadzu GC-14B) using a thermal conductive (TCD) and a flame ionization detector (FID). The temperature of the column (HayeSep Q, length 2 m) was  $140\text{ }^{\circ}\text{C}$ .

Photocatalytic experiments at solid-liquid interface were carried out in a 400 ml batch reactor, thermostated at  $25\text{ }^{\circ}\text{C}$ . For sample irradiation a 150 W power, immersion type high-pressure mercury lamp (Heraeus TQ 150) was used, surrounded with a glass filter in order to the high-energy photons ( $\lambda < 310\text{ nm}$ ) to be filtered out.

The concentration of phenol were determined by HPLC, with a UV-vis detector at  $\lambda = 210\text{ nm}$ . Phenol and its intermediates were separated on a C-18 column (LiChrospher 100 RP-18 ( $5\mu\text{m}$ )) using a mixture of acetonitrile/water (20/80) eluent with flow rate of  $1\text{ ml perc}^{-1}$ .

The degradation process in liquid phase was also followed by measuring the total carbon content (TOC) in the dispersions using Euroglas TOC 1200 apparatus.

## Novel scientific results

### I. Theses related to structural properties

#### 1. Synthesis and functionalization of titanium dioxide

Phosphate-modified titanium dioxide (henceforth abbreviated as P-TiO<sub>2</sub>) was prepared by treatment of TiO<sub>2</sub>·nH<sub>2</sub>O with phosphoric acid. The sol-gel synthesis method applied is divided into two main steps: (i) In the course of the reaction of titanium(IV)-isopropoxide and water, amorphous titanium oxide hydroxide (TiO<sub>2</sub>·nH<sub>2</sub>O) is formed by hydrolysis of the precursor and subsequent condensation of the product. (ii) TiO<sub>2</sub>·nH<sub>2</sub>O reacts with phosphoric acid, and phosphate species are chemisorbed on the surface of oxide nanoparticles. The phosphate content of P-TiO<sub>2</sub> does not decrease by either washing with water or calcination at 700 °C.

Phosphate species can be removed from the surface of P-TiO<sub>2</sub> by treatment in sodium hydrogen carbonate solution.

#### 2. Distribution and chemical environment of phosphorus in the surface region of P-TiO<sub>2</sub> and comparison of surface and bulk compositions

2.1. X-ray photoelectron spectroscopy showed that the oxidation state of titanium both in unmodified TiO<sub>2</sub> and in P-TiO<sub>2</sub> was +4.

2.2. Deconvolution of the O 1s region of the oxide samples allows distinction of four types of bonds involving oxygen: Ti–O–Ti, Ti–O–P, H–O–P, and H–O–C. When the P/Ti atomic ratio is systematically increased, the number of Ti–O–Ti bonds decreases, whereas the abundance of Ti–O–P and H–O–P bonds simultaneously increases. In the course of phosphoric acid treatment, the surface region of titanium dioxide is partly or entirely converted to titanium(IV)-phosphate, depending on the P/Ti ratio.

2.3. Phosphorus is always present in an O–P environment in P-TiO<sub>2</sub> samples; P–Ti bonds are not detectable.

2.4. Quantitative analysis of the XP spectra of sample P-TiO<sub>2</sub>/0.05 recorded at various angles did not reveal any changes in the P/Ti atomic ratio. Taking into account the escape depth of the P 2p electrons originating from the sample, we concluded that the estimated thickness of the phosphate layer covering the surface of the functionalized oxide is larger than 3 nm.

2.5. Determination of the titanium and phosphorus contents of the P-TiO<sub>2</sub> samples (ICP-AES) revealed that in the atomic ratio range of P/Ti= 0.01-0.30 the entire amount of

phosphoric acid added during synthesis was chemisorbed on amorphous  $\text{TiO}_2 \cdot n\text{H}_2\text{O}$ . Comparison of the results obtained by ICP-AES and the surface-sensitive XPS allowed to conclude that the surface region of P- $\text{TiO}_2$  samples is considerably enriched in phosphate as compared to the bulk phase. Surface accumulation of phosphate decreases with increasing P/Ti atomic ratio.

2.6. XPS measurements evidence that, a single treatment in  $\text{NaHCO}_3$  solution reduces the amount of phosphorous of the P- $\text{TiO}_2$  in the surface region by  $\sim 75\%$ .

### **3. The crystalline structure of P- $\text{TiO}_2$ and changes in crystallite size with phosphate content**

3.1. Transitions taking place upon calcination of amorphous titanium oxide samples are (i) the amorphous  $\text{TiO}_2 \cdot n\text{H}_2\text{O} \rightarrow$  anatase transition and (ii) the anatase  $\rightarrow$  rutile phase transition depending on the actual temperature. It can be established from X-ray diffraction patterns that the anatase content of bare titanium dioxide increases up to 500 °C. However, between 500 and 700 °C the amount of anatase decreases because it is transformed to rutile.

3.2. It was shown that in P- $\text{TiO}_2$  samples – unlike in the unmodified oxide – the transformation of amorphous  $\text{TiO}_2 \cdot n\text{H}_2\text{O}$  to anatase is inhibited and anatase content decreases in a linear fashion with increasing phosphate content. X-ray diffraction measurements also revealed that in P- $\text{TiO}_2$  samples the anatase  $\rightarrow$  rutile transformation was also inhibited up to 900 °C. Inhibition of the process  $\text{TiO}_2 \cdot n\text{H}_2\text{O} \rightarrow$  anatase  $\rightarrow$  rutile, observed during calcination of P- $\text{TiO}_2$  is due to the stabilizing effect of surface Ti–O–P bonds forming upon functionalization of  $\text{TiO}_2 \cdot n\text{H}_2\text{O}$ .

3.3. Phosphoric acid treatment of  $\text{TiO}_2 \cdot n\text{H}_2\text{O}$  yields amorphous titanium(IV)-phosphate, which is transformed to crystalline  $\text{TiP}_2\text{O}_7$  after calcination at 900 °C.

### **4. The thermal stability of P- $\text{TiO}_2$**

4.1. Nitrogen sorption experiments show that the specific surface area of unmodified  $\text{TiO}_2$  significantly decreases with increasing calcination temperature. The unmodified oxide has mesoporous structure up to 500 °C, which collapses when heated to higher temperatures.

4.2. The thermal stability of P- $\text{TiO}_2$  depends on the P/Ti atomic ratio. At  $\text{P/Ti} < 0.10$  the samples are mesoporous even after calcination at 700 °C and their specific surface area shows

a less pronounced with increasing calcination temperature than can be observed in the case of the unmodified oxide. At atomic ratios over  $P/Ti > 0.1$  the thermal stability of the samples decreases and after calcination at 700 °C they become non-porous.

## **5. Changes in optical properties (band gap energy) and photoconductivity as a function of phosphate content and calcination temperature**

5.1 Diffuse reflection UV-vis spectroscopy proves that band gap energy ( $E_g$ ) decreases with increasing calcination temperature both for unmodified and phosphate-modified titanium dioxides. The decrease in  $E_g$  observed in the course of calcination is due to an increase in the size of anatase crystallites and, depending on the phosphate content of the samples, to the anatase  $\rightarrow$  rutile transformation. Since both of these processes are inhibited in the P-TiO<sub>2</sub> samples, the band gap energies of these samples are higher than that of the unmodified oxide at all calcination temperatures.

5.2 The photoconductivity of titanium dioxide and P-TiO<sub>2</sub> thin layers ( $t = 2.8 \pm 0.2 \mu\text{m}$ ) was studied on interdigitated electrodes. It was shown that the oxide films exhibited nearly ohmic behaviour. During UV illumination the photocurrent can be measured reproducibly. It was also established that the photoconductivity of P-TiO<sub>2</sub> layers significantly decreased with increasing phosphate content, owing to the smaller particle size and the trapping of the photogenerated charge carriers by phosphate species. The latter is confirmed by the observation that elimination of surface phosphate species of P-TiO<sub>2</sub> samples by treatment with NaHCO<sub>3</sub> solution results in a ~40-fold increase in photocurrent. Thus, photoconductivity measurements indicate that recombination of photogenerated electrons and holes are inhibited by phosphate species.

## **II. Photoactivity of P-TiO<sub>2</sub> in solid/gas and solid/liquid interfaces**

Substrate conversion rates on P-TiO<sub>2</sub> catalysts are affected by phosphate modification of TiO<sub>2</sub>·nH<sub>2</sub>O. First, the amount of phosphate (the P/Ti atomic ratio applied) determines structural (the amount of photoactive anatase formed in the course of calcination, the particle size and the specific surface area of the oxide) and sorption properties of the photocatalyst. Furthermore, the chemisorbed phosphate promotes charge transfer at the interface by trapping photogenerated charge carriers and by inhibiting their recombination. Considering these issues the following conclusions can be drawn:

## **6. Photooxidation of ethanol at the solid/gas interface**

6.1. It was shown that the rate of photooxidation of ethanol, which has high affinity for adsorption, is the highest on P-TiO<sub>2</sub> catalysts with high specific surface area and anatase content. These criteria are accomplished in the composition range of P/Ti = 0.05-0.10.

6.2. It was established that water at relative humidities over 50 % significantly increased the amount of ethanol adsorbed as well as the rate of photooxidation on P-TiO<sub>2</sub> catalysts. It was revealed that in the presence of water both the conversion of acetaldehyde, the main intermediate of the reaction, and the rate of complete mineralization increased significantly.

6.3. After removal of surface phosphate species by sodium hydrogen carbonate, the amount of ethanol adsorbed on the catalyst surface increases, although the rate of photooxidation decreases significantly. This indicates that the charge carriers have a reduced life time in the absence of surface-bound phosphate.

6.4. After removal of the surface phosphate, neither the amount of adsorbed ethanol nor the rate of its photooxidation depend significantly on the presence of water in contrast to the modified catalysts.

## **7. Photooxidation of phenol at the solid/liquid interface**

7.1. In the case of the photooxidation of phenol, which adsorbs less readily than ethanol, the anatase content of the photocatalyst dominates over specific surface area. Since the anatase content decreases with phosphate content in a nearly linear fashion, the P-TiO<sub>2</sub> sample with the lowest phosphate content (P/Ti=0.01) proved to be the most efficient photocatalyst. At atomic ratios over P/Ti > 0.01 the rate of phenol degradation decreases considerably.

7.2. After removal of surface phosphates by sodium hydrogen carbonate, the amount of phenol adsorbed on the catalyst surface increases. However, the initial rate of its photooxidation decreases, suggesting that charge separation is also promoted by phosphate species at the solid/liquid interface.

7.3. After removal of surface phosphates the rate of the complete mineralization of phenol increases considerably and lower amounts of intermediates are accumulated during photooxidation. Comparison of these results with the above theses allows to conclude that the mechanism of phenol conversion on P-TiO<sub>2</sub> differs from that on the surface of TiO<sub>2</sub>.



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**ΣIF = 29.02**

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