Ph.D. Thesis

Decontamination of environmental pollutants by UV- and visible light-active titanium dioxide-based photocatalysts

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1. INTRODUCTION AND AIMS OF THE RESEARCH

Heterogeneous photocatalysis is nowadays an intensively investigated research topic. The production and behavior of visible light-active titanium dioxide-based photocatalysts are investigated in many publications. These photocatalysts can be applied in economic water purification processes, in which solar irradiation is applied, and also for the preparation of indoor self-cleaning/air-cleaning surfaces. The immobilization of the photocatalysts is a crucial question in these processes. The aims of my research were to investigate essential aspects of the practical applicability of heterogeneous photocatalysis: the production of differently modified, visible light active, titanium dioxide based photocatalysts, possible immobilization methods, the detailed characterization of these photocatalysts, and their comparison with commercial photocatalysts.

For the determination of the particle sizes and the crystal phase distributions of the investigated titanium dioxides, X-ray diffraction (XRD) measurements were carried out. The dopant contents of the modified titanias were measured by X-ray photoelectron spectroscopy (XPS). The specific surface areas were determined by nitrogen adsorption. For the specification of the light absorption of the photocatalysts, diffuse reflectance spectroscopy (DRS) was applied. Pictures of the photocatalyst nanoparticles were taken by transmission electron microscopy (TEM) for determination of the size distributions and shapes of the nanoparticles. The photocatalytic activities were determined with different light sources (UV, VIS and solar irradiations) and different model pollutants (phenol and *Escherichia coli* bacteria).

Connections were explored between the photocatalytic efficiencies of different titanium dioxides and the photocatalytically produced reactive species by electron spin resonance (ESR) measurements.

The photocatalytic efficiencies of the investigated photocatalysts were characterized in outdoor photocatalytic experiments, in which solar irradiation was applied for the activation of the photocatalysts. The wavelength dependence of the photocatalytic efficiency of the promising titanium dioxides was investigated in details.

Attempts were made to develop different methods for the immobilization of the photocatalysts, and the produced photocatalytically active, durable surface was applied in a self-designed and home-made, fixed-bed, recirculating flow reactor. A further aim was to produce a pilot-scale photoreactor in which the developed photocatalytically active and durable surface was utilized. This photoreactor can provide the basis for a mobile, water purifier apparatus, which needs only solar light for operation.
2. METHODS

2.1. Determination of photocatalytic efficiencies

For the determination of photocatalytic efficiencies, home-made photoreactors were applied, which were equipped with UV- (6 of Vilber-Lourmat T-6L UV-A type, 6 W fluorescent tubes), or visible light-emitting (4 of Düwi 25920/R7S type, 24 W energy-saving, compact fluorescent tubes) light sources. In some cases, different colored 5050 SMD LED strips (14.4 W) were applied for the activation of the photocatalysts.

2.2. Determination of disinfection efficiencies

The photocatalytic efficiencies of the investigated photocatalysts were also determined by the inactivation of Escherichia coli K12 bacteria in some cases. The experiments were carried out in the photoreactor, which was equipped with visible light-emitting light sources. The numbers of the colony forming units of the treated waters were determined by counting the grown colonies (on agar-agar gels) from the taken samples.

2.3. Flow reactor equipped with immobilized photocatalysts

The produced photocatalytically active surfaces were applied in a self-designed, home-made, fixed-bed, recirculating flow reactor. For the activation of the immobilized photocatalysts, UV fluorescent tubes (Lightech UVA; 4×40 W) or visible light-emitting reflectors (Jen CE-82; 2×500 W) were applied.

2.4. Liquid chromatography

The concentrations of the model contaminants were determined by high-performance liquid chromatography (HPLC) with Agilent 1100 series equipment.

2.5. Electron spin resonance (ESR) measurements

For the investigation of the reactive species produced by the activated photocatalysts, ESR measurements were carried out with a Bruker Biospin ESP300E spectrometer. The reactive oxygen species scavengers applied were 2,2,6,6-tetramethyl-4-piperidinol (TMP-OH) and 5,5-dimethyl-1-pyrroline N-oxide (DMPO). In some cases, heavy water and sodium azide were also used.

2.6. Determination of photon fluxes

The photon fluxes in the applied photoreactors were determined by ferrioxalate actinometry, which is one of the most commonly used light intensity determination methods in photochemistry.

In the case of the light sources, when $\lambda > 550$ nm, the light intensity was determined with an Apogee MQ-200 PPF (photosynthetic photon flux)-meter.
2.7. Characterization of the photocatalysts

Pictures used for the characterization of the shape, the size distribution and (in some cases) the size of the nanoparticles were taken with a Philips CM 10 (100 kV) transmission electron microscope.

XRD measurements were carried out with a Rigaku Miniflex II diffractometer ($\lambda_{\text{Cu K}\alpha} = 0.15406$ nm, 40 kV and 30 mA).

The DR spectra of the samples were determined with a JASCO-V650 diode array spectrophotometer, equipped with an ILV-724 DR module ($\lambda = 220-800$ nm; resolution: 0.5 nm; scanning speed: 100 nm/min).

The specific surface areas of the photocatalysts were measured by nitrogen adsorption at 77 K, using a Micromeritics gas adsorption analyzer (Gemini Type 2375). The specific surface areas were calculated via the BET method.

For the investigation of the dopant contents and the surfaces of the photocatalysts, XPS was applied. For the measurements, a Specs spectrometer was used, equipped with a Phoibos 150 MCD 9 electron analyzer. The X-ray photoelectron source was the K$_\alpha$ radiation of a Mg anode ($h\nu = 1253.6$ eV).

The FT-IR measurements were made with a Bruker Equinox 55 spectrometer with an integrated FRA 106 Raman Module. Samples were ground with KBr and pressed into thin pellets (thickness $\approx 0.3$ mm), and IR spectra were recorded with a spectral resolution of 2 cm$^{-1}$ in the 400-4000 cm$^{-1}$ region.

A Horiba Jobin Yvon XGT-5000 X-ray fluorescent spectrometer, equipped with a Rh X-ray source, was used to measure the element contents of some photocatalyst samples. The records were made at 30 kV excitation voltage, 0.5 mA anode current and 1000 s measuring time.
3. NOVEL SCIENTIFIC RESULTS

1. Gold and silver-containing TiO$_2$/noble metal nanocomposites do not have higher photocatalytic efficiency than the basic photocatalyst in the cases of phenol and *E. coli* model pollutants [1, 2].

XRF measurements proved that a TiO$_2$/Au nanocomposite (containing 0.96 wt% gold) and a TiO$_2$/Ag nanocomposite (containing 0.94 wt% silver) were successfully synthetized. In the case of phenol as model contaminant, and UV irradiation, the noble metal-containing titanium dioxide nanocomposites showed slightly lower photocatalytic performances than that of the basic titanium dioxide [1]. In the case of visible light irradiation, the TiO$_2$/Ag nanocomposite displayed similar efficiency to that of the basic titanium dioxide, while the TiO$_2$/Au nanocomposite did not exhibit significant photocatalytic activity [2]. In the case of *E. coli* as model contaminant (only visible light irradiation was investigated), the TiO$_2$/Ag nanocomposite showed similar efficiency to that of the basic titanium dioxide, while the TiO$_2$/Au nanocomposite did not exert any disinfection property [2]. On the basis of these results, it can be concluded that, for the decontamination of the investigated model contaminants, it is not appropriate to deposit the applied titanium dioxide with the investigated noble metals. It should be noted that with oxalic acid as model contaminant, and UV irradiation, the TiO$_2$/noble metal nanocomposites give an excellent photocatalytic performance [1], as it was also described in 2003 by Szabó-Bárdos *et al.* [3].

2. Under visible light irradiation, those photocatalysts exert photocatalytic disinfection property, which generates hydroxyl radical [2]. The photocatalytic disinfection efficiency of the prepared, iodine-doped titanium dioxide was not caused exclusively by photocatalytic effects [4].

The results of photocatalytic experiments with phenol and *E. coli* as model contaminants, and the results of ESR measurements are presented in Table 1. It can be seen that the titanium dioxides that displayed disinfection properties under visible light irradiation, also generated hydroxyl radicals. Those titanium dioxides which did not generate hydroxyl radicals, did not exhibit a disinfection effect; however, in some cases these titanium dioxides demonstrated a very high photocatalytic performance in the case of phenol decomposition.
### Photocatalytic performances and the results of the ESR measurements

Iodine-doped titanium dioxide was synthetized by Hong et al. [5] by the dropwise addition of titanium(IV) butoxide into iodic acid solution (c = 0.15 M). In my research a series of iodine-doped titanium dioxides were synthetized with different n\textsubscript{I}/n\textsubscript{Ti} ratios (0.0, 0.1, 0.5, 1.3 and 2.6) [4]. The photocatalyst with n\textsubscript{I}/n\textsubscript{Ti} = 0.5 had the highest photocatalytic activity for both UV and VIS irradiation. XPS measurements proved that this titanium dioxide has 0.67 at% iodine on the surface of the particles, in the form of I\textsuperscript{-} (61%) and I\textsuperscript{7+} (39%). The generation of elemental iodine in the visible light-irradiated suspension, which can contribute to the high disinfection performance of this titanium dioxide was proved by spectrophotometry [4].

3. Non-doped, rutile-phase titanium dioxide with small particle size was synthetized via the HCl-promoted hydrolysis of titanium(IV) butoxide followed by crystallization at low temperature (40 °C). The calcination of this titanium dioxide with increasing temperature (400-1000 °C) resulted in increasing particle size, increasing absorption of visible light and decreasing specific surface area [6]. The photocatalytic activity of the home-made rutile (calcinated at 900 °C) was much lower than that of Aldrich rutile, even though these titanium dioxides have very similar structural properties [6].

For the preparation of rutile-phase titanium dioxide with small particle size, the preparation method described by Tang et al. [7] was modified. The HCl hydrolysis of titanium(IV) butoxide at a molar ratio Ti(OC\textsubscript{4}H\textsubscript{9})\textsubscript{4}:H\textsuperscript{+}:H\textsubscript{2}O = 1:3:50 resulted in pure rutile-phase titanium dioxide [6]. With the utilization of HCl, possible nitrogen incorporation can be excluded.

<table>
<thead>
<tr>
<th>Photocatalysts</th>
<th>r\textsubscript{0,phenol} (×10\textsuperscript{-8} M/s)</th>
<th>t Sterilization (min)</th>
<th>ESR measurements with TMP-OH as scavenger</th>
<th>ESR measurements with DMPO as scavenger</th>
<th>ESR measurements with DMPO as scavenger</th>
</tr>
</thead>
<tbody>
<tr>
<td>TiO\textsubscript{2}-VLP7000</td>
<td>29.9</td>
<td></td>
<td>High</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>TiO\textsubscript{2}-I</td>
<td>5.0</td>
<td>20</td>
<td>-</td>
<td>-</td>
<td>Yes</td>
</tr>
<tr>
<td>TiO\textsubscript{2}-AR</td>
<td>4.2</td>
<td>20</td>
<td>-</td>
<td>-</td>
<td>High</td>
</tr>
<tr>
<td>TiO\textsubscript{2}-P25-NS</td>
<td>3.7</td>
<td>-</td>
<td>Not measured</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>TiO\textsubscript{2}-N</td>
<td>2.4</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>TiO\textsubscript{2}-Fe</td>
<td>1.7</td>
<td>-</td>
<td>Not measured</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>TiO\textsubscript{2}-TP-S201</td>
<td>1.5</td>
<td>60</td>
<td>Not measured</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>TiO\textsubscript{2}-P25</td>
<td>1.4</td>
<td>60</td>
<td>-</td>
<td>-</td>
<td>Yes</td>
</tr>
<tr>
<td>TiO\textsubscript{2}-P25-Ag</td>
<td>1.3</td>
<td>60</td>
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<tr>
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<td>-</td>
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<td></td>
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<tr>
<td>TiO\textsubscript{2}-AA</td>
<td>0.4</td>
<td>-</td>
<td>Not measured</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 1.
XRF and TEM measurements proved that rutile-phase titanium dioxides (Table 2) with increasing particle sizes can be prepared by calcination (with increasing temperature: 400-1000 °C) of home-made nano rutile (average particle size: 5.2 nm; specific surface area: 197 m²/g).

<table>
<thead>
<tr>
<th>Photocatalyst</th>
<th>Phase distribution</th>
<th>Specific surface area (m²/g)</th>
<th>R₀, phenol (10¹² mol/m²/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Anatase</td>
<td>Rutile</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Content (wt%)</td>
<td>Particle size (nm)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Content (wt%)</td>
<td>Particle size (nm)</td>
<td></td>
</tr>
<tr>
<td>Rutile - O</td>
<td>-</td>
<td>100</td>
<td>5.2</td>
</tr>
<tr>
<td>Rutile - RHSE-400</td>
<td>-</td>
<td>100</td>
<td>12.9</td>
</tr>
<tr>
<td>Rutile - RHSE-600</td>
<td>&lt;1</td>
<td>&gt;99</td>
<td>39.1</td>
</tr>
<tr>
<td>Rutile - RHSE-700</td>
<td>&lt;1</td>
<td>&gt;99</td>
<td>69.3</td>
</tr>
<tr>
<td>Rutile - RHSE-800</td>
<td>&lt;1</td>
<td>&gt;99</td>
<td>135 TEM</td>
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<tr>
<td>Rutile - RHSE-900</td>
<td>&lt;1</td>
<td>&gt;99</td>
<td>245 TEM</td>
</tr>
<tr>
<td>Rutile - RHSE-1000</td>
<td>&lt;1</td>
<td>&gt;99</td>
<td>290 TEM</td>
</tr>
<tr>
<td>Aldrich rutile</td>
<td>4</td>
<td>315 TEM</td>
<td>96</td>
</tr>
<tr>
<td>Aerioxide P25</td>
<td>90</td>
<td>25.4</td>
<td>10</td>
</tr>
</tbody>
</table>

Table 2.

Phase distributions, particle sizes, specific surface areas and photocatalytic efficiencies

The visible light absorption of the synthetized photocatalysts increased in the sequence of increasing calcination temperature, as can be seen in Figure 1, which shows the red-shifted light absorption onset (measured by DRS) of the photocatalysts.

![Figure 1.](image)

Light absorption of the investigated photocatalysts (DRS)
The photocatalytic efficiency slightly decreased in the sequence of increasing calcination temperature because of the combined effect of the decreasing specific surface area and the decreasing visible light absorption. However, the surface-normalized photocatalytic efficiency decreased strongly in the same sequence.

The commercial (Aldrich) rutile has very similar structural properties to those of the self-prepared Rutile-RHSE-900 photocatalyst, but it has significantly higher photocatalytic efficiency. The only difference between the IR spectra of these photocatalysts is the appearance of a new band for Aldrich rutile at 687 cm\(^{-1}\), which indicates Ti-O-O-Ti groups \([8, 9]\) on the surface of this titanium dioxide. These groups result in an oxygen-rich surface, which could be an electrophilic entity. This “peroxidized” surface can attract electrons, which can then be easily captured by molecular oxygen, resulting in a higher rate of radical generation.

4. In the case of rutile-phase titanium dioxide, the presence of Ti\(^{3+}\) and low-binding-energy oxygen (which indicates defects) have no significant importance for the photocatalytic performance under visible light irradiation \([6]\) (in contrast with the UV-irradiated, anatase-phase titanium dioxides \([10]\)).

In the Ti2p XPS spectrum of Aldrich rutile, only Ti\(^{4+}\) was detected, while the O1s spectrum revealed the usual components: oxide oxygen from titanium dioxide (530.3 eV), surface OH group oxygen (532 eV), and oxygen from H\(_2\)O (532.8 eV) \([6]\). In the case of the self-prepared Rutile-RHSE-900, the Ti2p XPS spectrum demonstrated a small amount of Ti\(^{3+}\) (13 at\%: peaks at 457.3 eV and 461.9 eV), along with Ti\(^{4+}\) (87 at\%: peaks at 459.1 eV and 464.8 eV). The O1s spectrum of this material indicated (besides the usual oxygen signals) a low-binding-energy oxygen species (at 528.8 eV, 12 at\%). On the basis of recent publications \([10, 11]\), this interesting form of oxygen indicates defects, or oxygen atoms adjacent to Ti\(^{3+}\). In the case of anatase-phase titanium dioxides, the presence of this special species enhanced the photocatalytic activity \([10]\) under UV irradiation (for phenol degradation). Nevertheless, Rutile-RHSE-900 (which contains Ti\(^{3+}\) and the described low-binding-energy oxygen species) has significantly lower photocatalytic efficiency than Aldrich rutile (under visible light irradiation) \([6]\).

5. The intensity of commercially available lamps in the wavelength range from 400 nm to 420 nm is crucial for the effective application of photocatalysts in indoor air/surface cleaning processes \([2]\).

Photocatalytic disinfection experiments (with \textit{E. coli} bacteria) were also carried out with K\(_2\)Cr\(_2\)O\(_7\) (5 mM) light filtration, which resulted in reduction of the light intensity under 420 nm to
4% of that in the case of NaNO₂ (1 M) light filtration. With this reduced light intensity, three photocatalysts (which sterilized the treated water after 1 h of irradiation in the case of NaNO₂ light filtration) lost their disinfection effect, and in the case of two other titanium dioxides (which sterilized the treated water after 20 min of irradiation in the case of NaNO₂ light filtration) the disinfection efficiency was also significantly reduced with K₂Cr₂O₇ light filtration [2]. These results clearly showed that the light intensity (in the wavelength range between 400 and 420 nm) of the applied light sources is crucial for indoor air/surface cleaning processes.

6. In solar light-based, photocatalytic water treatment processes, the utilization of non-doped, anatase-phase titanium dioxides should be preferred [12].

The photocatalytic decomposition of phenol was additionally investigated in the case of solar irradiation, and it was concluded that the three non-doped, mainly anatase-phase titanium dioxides (which have low, if any activity under visible light irradiation), give significantly higher photocatalytic performances than those of highly visible light-active titanium dioxides.

For the explanation of these results, the wavelength dependence of the photocatalytic efficiency of the titanium dioxides was investigated (Figure 2).

![Figure 2. Wavelength dependence of the photocatalytic efficiency of titanium dioxide samples](image)

In the case of UV irradiation, the non-doped, mainly anatase-phase titanium dioxides (TiO₂-LH, Aeroxide P25 and Aldrich anatase) revealed significantly higher photocatalytic performances than those of doped and/or rutile-phase titanium dioxides. Under visible light irradiation, the doped or rutile-phase titanium dioxides displayed better performances. This is clearly seen in the case of
violet irradiation, but at longer wavelengths only the commercial (KRONOS) TiO$_2$-VLP7000 photocatalyst exhibited significant photocatalytic efficiency. Although this latter photocatalyst can be activated by photons throughout the whole visible light range, the apparent quantum yields in most of the visible light range are two orders of magnitude smaller, than the apparent quantum yields of non-doped, mainly anatase-phase titanium dioxides under UV irradiation.

7. Crystallized titanium dioxide nanoparticles can be immobilized into an amorphous titanium oxide-hydroxide layer, while the photocatalysts maintain their photocatalytic property. The photocatalytically active surface produced is not sensitive to UV irradiation or to the oxidative effect of activated titanium dioxide nanoparticles [13].

A sheet of ceramic paper was perfused with ethanol, and was then impregnated with titanium(IV) ethoxide. After this step, precrystallized photocatalysts were immediately sprayed in suspension (ethanol) form with a handheld airbrush onto the surface (Figure 3).

![Figure 3](image)

*Figure 3.*

_Schematic figure of the method of immobilization of titanium dioxide nanoparticles_

The impregnated ceramic sheets were dried in air at room temperature for 24 h. During this step, the titanium(IV) ethoxide was hydrolyzed by the air humidity, and the amorphous Ti(IV) oxide-hydroxide formed fixed the photocatalytically active particles onto the surface. After the drying step, the impregnated ceramic sheets were washed with distilled water to eliminate non-
immobilized nanoparticles, and were finally cleaned from possible organic surface contaminants by irradiation with UV-A light for 24 h.

The photocatalytic performance of the prepared photocatalytically active surface was well reproduced. The differences in photocatalytic efficiency of three different immobilized titanium dioxide-coated ceramic papers were < 1%. The durability was confirmed in photocatalytic experiments in which the rate of phenol decomposition remained constant throughout five 2-h cycles on the same titanium dioxide-coated ceramic paper. Outdoor solar experiments were carried out to demonstrate that organic contaminants can be decomposed by solar light with the immobilized titanium dioxide, and we produced a pilot-scale photoreactor which requires only solar light for the purification of contaminated water.

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  Papers related to the Thesis: 4 (Cumulative impact factor: 16,164)
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Conferences: 35
  as speaker: 21
  as co-author: 14
Book chapters: 7

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