The synthesis of TiO_2/WO_3 composite photocatalysts - the role of the WO_3 in the charge trapping process

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Doctoral (Ph.D.) thesis

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Introduction

Nowadays, environmental issues are increasingly highlighted and studied worldwide. Numerous publications, forums, conferences deal with these environmental issues concerning the pedosphere, the atmosphere and hydrosphere. One of the main problems are water resources, which are constantly decreasing and deteriorating in quantity and quality.The Environmental Chemistry Research Group from the University of Szeged is working on the development of innovative water purification technologies, which are implemented through the advanced oxidation processes. One of these processes is heterogeneous photocatalysis, in which the photocatalyst generates electron-hole (e^- and h^+) pairs on its surface. The photogenerated charge carrier pairs can participate in redox reactions, although their recombination is a favored process.

Titanium dioxide (TiO_2) is one of the most widely used photocatalysts in the UV range [1-3]. The studied materials, the photocatalysts were used in many interesting and useful applications such as: organic pollutant degradation, like phenol [4], oxalic acid [5-6]; exploitation of the solar energy [7], like dye–sensitized solar cells [8], photocatalytic H_2 production [9-11], etc. Besides titania photocatalysts, there are other semiconductors which are applied in photocatalysis, for example WO_3 or ZnO. The main advantage of tungsten oxide is the high stability and lower band-gap energy. The latter means that the photocatalysts can absorb the sunlight more efficiently (from visible to UV). Taking advantage of the above listed properties, WO_3 can be applied as a photoelectrocatalyst. During the preparation of TiO_2/WO_3 composite photocatalysts, we focused on achieving efficient electron-hole separation and delayed recombination of charges.

Objectives of the present PhD. thesis

The main objective is to obtain and to characterize TiO_2/WO_3 composite photocatalysts, while focusing on the charge separation properties of WO_3. The composites will be obtained using commercial TiO_2 (Evonik Aeroxide P25, Aldrich anatase and rutile), while different synthesis approaches will be also investigated, such as ultrasonic and physical mixing, chemical reduction (noble metals) and impregnation. These methods would give information concerning the role of the WO_3 component (crystal geometry, morphology, crystal structure, etc.). Other charge separators will be applied to enhance further the photoactivity of the composites.

In the case of the composites obtained by ultrasonic homogenization it will be investigated, that:

- the presence of additional composite components, *such as MWCNT* can influence the activity of the material,
- if yes, *what is the impact if its concentration* in the composite

When selective photoreduction was applied to obtain TiO_2/WO_3 /noble metal ternary composites, it will be investigated:

- the importance of *the noble metal nanoparticles' localization* on a specific oxide,
- *the impact of the WO_3 content* on the photoactivity of the composites

The impregnation approach will be investigated as alternative approach in obtaining WO_3 forms, influencing positively the activity, when:

- *the WO_3 content dependence* will be investigated of the different WO_3 forms and their general impact on the photoactivity of the composites.
- is the photoreduction of noble metals by different light sources important?

In case of simple physical mixing of the components, the following important parameters will be investigated which may have major importance in defining the photoactivity of the composites: crystal structure and morphology trough shape tailoring approaches

Synthesis of the TiO_2/WO_3 composites

Synthesis of the TiO_2/WO_3 composites by reduction and ultrasonication

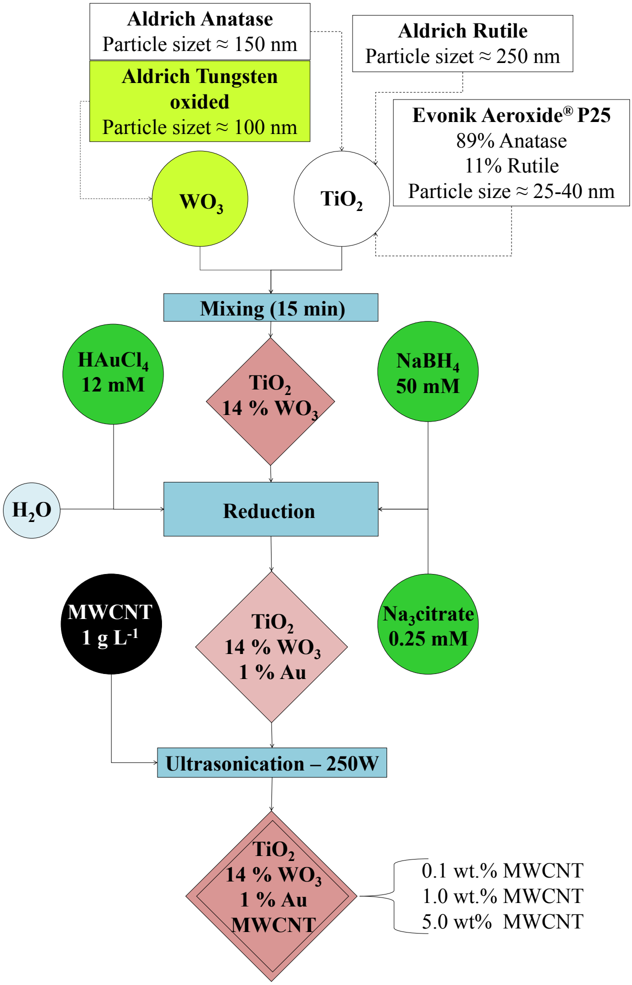

Figure 1. Synthesis of the $TiO_2/WO_3/Au/MWCNT$ composites

Synthesis of the TiO_2/WO_3 composites by selective deposition

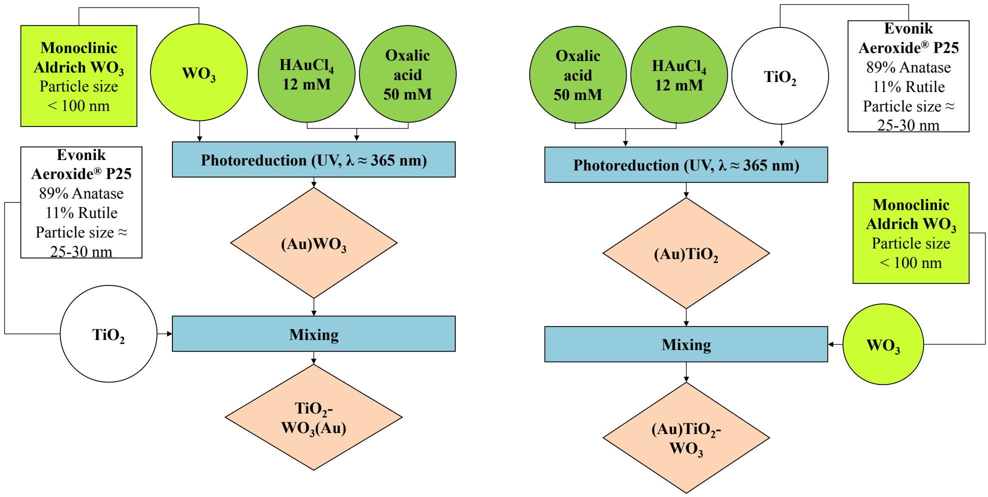

Figure 2. Synthesis of the TiO_2/WO_3 composites by selective photodeposition

Synthesis of the TiO_2/WO_3 composites by impregnation

All chemicals used in synthesis of the composites were of analytical grade (from Sigma-Aldrich) and were used without further purification. 0.78 g (or 0.13 g in the case of the catalysts with 4 wt. % WO_3) of H_2WO_4 (as WO_3 precursor) was added to 150 mL 6.6 wt.% NH_3 aqueous solution, under stirring at room temperature. A specific amount (2.27 g – 4 wt.% WO_3 containing composites, 2.88 g – in the case of 24 wt.% WO_3 containing composites) of TiO_2 (Evonik Aeroxide P25) was transferred in the obtained solution with continuous stirring at 60 ºC for 30 min. The obtained composite was dried in an oven at 80 °C for 24 hours and calcined at 700 ºC in a muffle furnace (heating rate 4 ºC/min), for 2 hours in still-air.

The gold deposition: the gold was performed under UV light (6 × 6 W Black Light Lamps, with $\lambda_{max} \approx 365$ nm) using 12 mM $HAuCl_4$ solution as gold precursor and 50 mM oxalic acid as hole scavenger, in a thermostated 200 mL Pyrex reactor (filled with 175 mL suspension). The P25/WO_3 catalyst concentration was 1 g/L, while the gold precursor quantity was calculated to have an equivalent of 1 wt. % Au on the suspended composite. The reduction process was performed for 4 hours. The visible light deposition followed the procedure detailed previously, the only difference was that 6 × 6 W Philips fluorescent lamps (visible light emitting coating) were used and 1M $NaNO_2$ was circulated in the thermostating jacket to eliminate any UV radiations. All the composites were washed several times in deionized water and dried at 80 °C for 24 hours. Au content was proven to be ≈1 wt. % in all the composite materials and no traces of the precursor was detected using UV-Vis spectrophotometry.

Synthesis of the TiO_2/WO_3 composites using WO_3 crystals obtained via hydrothermal crystallization

Synthesis of the WO_3 crystals from AMT: WO_3 microcrystals were synthesized by hydrothermal crystallization. 0.664 g AMT (ammonium metatungstate) was used. The precursor was dispersed in 88.48 mL 5 M HCl solution and were continuously stirred for 20 minutes. After that 10 mg of fMWCNTs (functionalized multiwalled carbon nanotubes) were added as a crystallization's promoter (the fMWCNT content was constant in all the cases). This was followed by the addition of 1.52 mL of HF solution (40 v/v %). The mixture was poured into a 100 mL Teflon lined autoclave and maintained at 180 ºC for 1, 5 or 24 h. After it was left to cool down to room temperature, the product was washed (Eppendorf Centrifuge 5702; centrifuging time: 5 min, rev: 4400 rpm) and dried at 40 ºC for 24 h. *Synthesis of the WO_3 crystals from STD:* The process for the preparation of the WO_3 crystals produced from sodium tungstate is the same as for the production of WO_3 from AMT, except that the STD precursor was 0.858 g.

Methods and instrumentation

The *X-ray diffraction (XRD) of the patterns* were recorded using Cu-K_α radiation (λ=1.5406 Å) equipped with a graphite monochromator. The measurements were performed on a *Shimadzu 6000* diffractometer. The average size of the crystals was calculated using the Scherrer equation where it was possible.

JASCO-V650 spectrophotometer with an integration sphere (*ILV-724*) was used for measuring the DRS spectra of the samples (λ=300-800 nm).

SEM (Scanning Electronic Microscopy) micrographs were recorded using a *Hitachi S-4700 Type II* cold field emission scanning electron microscope equipped with a *Röntec QX2-EDS* spectrometer. *Transmission electron microscopic (TEM) measurements* were performed to characterize the crystallite size, distribution and to identify the morphology of the particles. TEM micrographs were recorded on a Philips CM 10 instrument operating at 100 kV using Formvar coated copper grids.

XPS measurements were performed on a SPECS PHOIBOS150 MCD instrument, with monochromatized Al K radiation (1486.69 eV) at 14 kV and 20 mA, and a pressure lower than10−9 mbar. Analysis of the obtained data was carried out with CasaXPS software.

The FT-Raman spectra were recorded by using a *Bruker Equinox 55 spectrometer* with an integrated *FRA 106 Raman* module using an Nd-YAG laser (1064 nm). Raman spectra were recorded with a spectral resolution of 1 cm^{-1}.

The assessment of the photocatalytic efficiencies

The photocatalytic activity was determined by the photodegradation of (i) 0.5 mM phenol and (ii) 5 mM of oxalic acid solution (the concentration values were chosen based on our long-term experience with these substrates. The concentration decrease of the chosen contaminant (phenol or oxalic acid) was followed using an *Agilent 1100 series HPLC* system. This consist of a binary pump; a micro vacuum degasser; a diode array detector ($\lambda_{detection}$ = (i) 210 nm (ii) 206 nm); a thermostated column compartment and ChemStation data managing software. The chromatographic system was equipped with Rheodyne Model 7725 injector with a 20 µL loop and a (i) Lichrosper PR 18 (ii) GROM-RESIN ZH column. The eluent consisted of (i) 8.8 M methanol aqueous solution (ii) 19.3 mM H_2SO_4 aqueous solution and the applied flow rate was 0.8 m · min^{-1} in all the cases.

Results and discussion. Key thesis points

Synthesis of the TiO_2/WO_3 composites by reduction and ultrasonication

T1: An appropriately low amount of MWCNT was favorable concerning the photocatalytic activity of the nanocomposites.

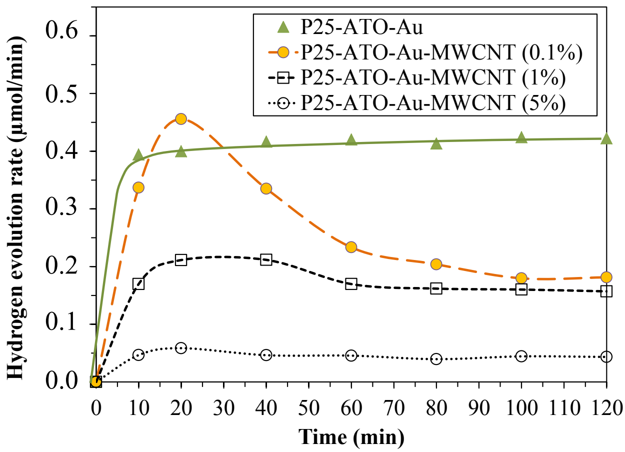

Figure 3. $TiO_2/WO_3/Au/MWCNT$ composites hydrogen production rate

P25-based composites showed a remarkable hydrogen production capacity compared to the pure anatase and rutile containing materials with large nanoparticles. The presence of MWCNT's lowered somewhat the hydrogen production rate. However, at low MWCNT concentration (0.1 wt.%) a small improvement was observed concerning the achieved maximum value of the hydrogen production rate (*Figure 3*), while at other MWCNT concentration values this enhancement was not observable (e.g. 0.1, 1.0 and 5.0 wt.%). The 0.1 wt.% value must be an optimum, as the photogenerated electrons can be captured either by WO_3, Au or even MWCNT, pointing out an intensive competition, between the electron-separators.

Synthesis of the TiO_2/WO_3 composites by selective photodeposition

T2: The localization of the noble metal nanoparticles within the composite was crucial.

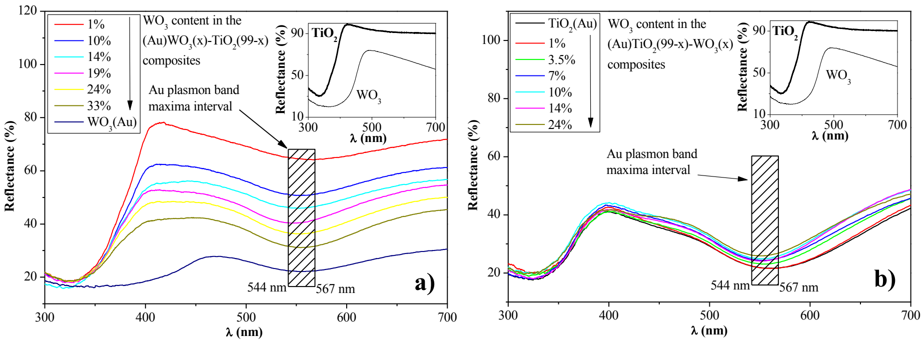

Figure 4. DRS spectra of sample series (a) (Au)WO_3(x)–TiO_2(99 − x) and (b) (Au)TiO_2(99 − x)–WO_3(x)

Considering the optical properties of the ternary composites, it was observed, that the reduction of gold on the surface of TiO_2, within the ternary composite resulted an optical independency (band-gap values) from the WO_3 content (*Figure 4b*). This was not the case when the Au was deposited on the surface of WO_3, namely that with the change of the WO_3 content, the band-gap of the composite changed likewise (*Figure 4a*). This points out a possibility to finely tune the band-gap of composites, using this approach. The observed optical difference was also valid for Pt, but no clear order or any trends were noticed.

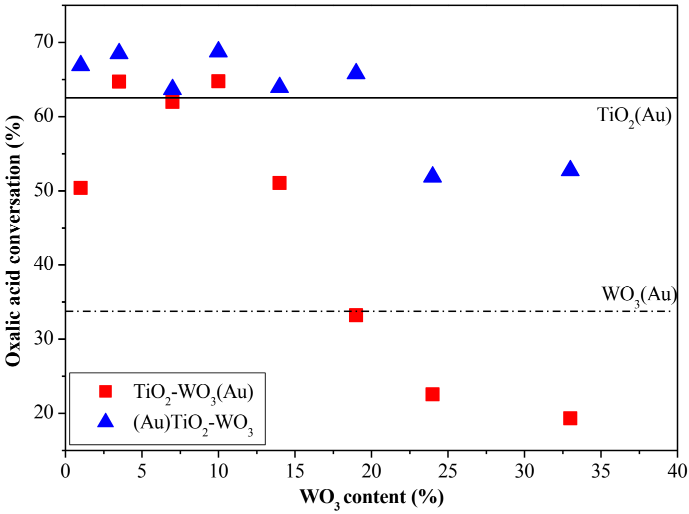

Figure 5. Photocatalytic performance of the sample series (Au)TiO_2(99 − x%)–WO_3(x%) and (Au)WO_3(x%)–TiO_2(99 − x%)

T3: The photocatalytic activity of the nanocomposites was maximum when the WO_3 concentration was around 10%.

With the increase of the tungsten (VI) oxide content the composites with noble metal deposited on TiO_2 showed different behavior compared to the ones with Au or Pt deposited on WO_3 (in the first case a roughly constant activity is observed, which decreases at higher WO_3 content, while in the latter case a bell-shaped curve is perceived with the maximum around 10 wt.% of WO_3). In all the series, at least two catalysts were surpassing the activity of the reference catalysts TiO_2(Au) and TiO_2(Pt), whilst nearly all the materials were more active than WO_3(Au) and WO_3(Pt) references (*Figure 5*). If the WO_3 content is raised above a critical level (24 wt.%) a severe activity decrease was observable in an of the studied cases, but the most spectacular decrease was observed, when the noble metals were deposited on the surface of WO_3.

Synthesis of the TiO_2/WO_3 composites by impregnation

T4: By choosing the impregnation as the synthesis pathway for the P25/WO_3 composites, resulted in a material which contained the three WO_3 forms (amorphous, crystalline, doped) in different proportions, which influenced critically the resulting photoactivity.

The obtained nanocomposites were investigated using different characterization techniques. When the DRS spectra were recorded and analyzed it was found that doping must occur in the composites which contained 4 wt.% of $WO_{3.}$

The Raman spectrum investigations pointed out, the previously mentioned aspect of doping, while specific signs of monoclinic WO_3 (710 and 800 cm^{-1}), and amorphous WO_3 (260 cm^{-1} (O-W-O), 950-960 cm^{-1} (W=O) and 1126 cm^{-1}) were registered, demonstrating the presence of both forms in the composites with just 4% of $WO_{3.}$ When the amorphous WO_3 was present, it was found that the photoactivity was enhanced, compared to the samples which contained 24% of $WO_{3.}$ The surface normalized reaction rate values of the most performing samples even surpassed the activity of Evonik Aeroxide P25 (*Table 1*)

Table 1. UV photodegradation rates of phenol and crystallite sizes

Sample	**r_{phenol} (µM min^{-1})**	**$r_{phenol,S}$ (mM h^{-1} m^{-2})**	**d (nm)**	
			TiO_2	**WO_3**
			Anatase/ Rutile	**Monoclinic**
$P25/WO_3(4\%)$	2.98	5.29	32/48	-
$P25/WO_3(4\%)$-Au-VIS	3.04	4.61	30/46	-
$P25/WO_3(4\%)$-Au-UV	3.92	5.95	30/46	-
P25	4.12	4.94	25/40	-
$P25/WO_3(24\%)$	1.10	0.83	31/43	22
$P25/WO_3(24\%)$-Au-VIS	0.78	0.67	32/44	24
$P25/WO_3(24\%)$-Au-UV	2.18	3.54	34/50	25

r_{phenol} – degradation rate of phenol; $r_{phenol,S}$ – surface normalized degradation rate of phenol; d – crystallite size

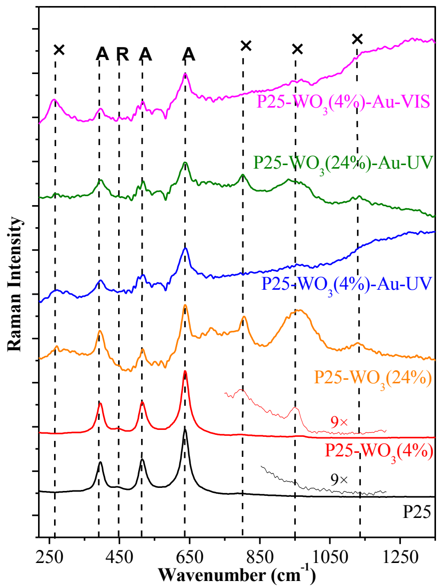

Figure 6. Raman spectra of the investigated photocatalysts

Synthesis of the TiO_2/WO_3 composites using WO_3 crystals obtained via hydrothermal crystallization

T5: The hydrothermal crystallization times affects the WO_3 crystals' size, morphology, crystal phase composition.

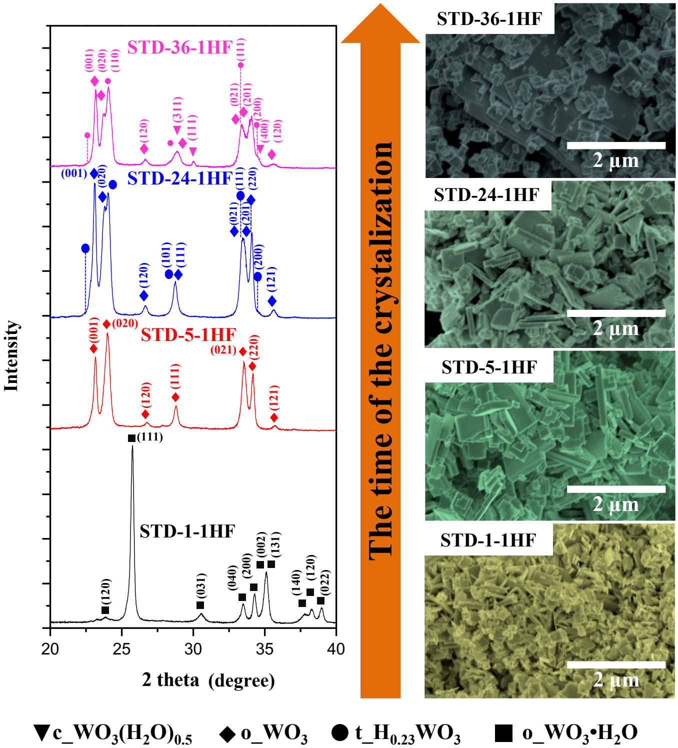

Figure 7. The XRD patterns and the crystallization time's influence on the WO_3 (obtained from STD) crystals' size

The crystal' size of the obtained samples increased with the duration of the crystallization time, while crystal phase changes were observed as well (orthorhombic

$WO_3 \cdot H_2O$ → orthorhombic WO_3 → tetragonal $H_{0.23}WO_3$ and tetragonal $WO_3(H_2O)_{0.5}$) (*Figure 7*). This proves, that by the simple adjustment of the crystallization time the crystal phase composition and chemical composition of the samples can be achieved successfully.

T6: The HF was responsible in the stabilization of certain crystallographic planes and thus for the geometry of the nanocrystals.

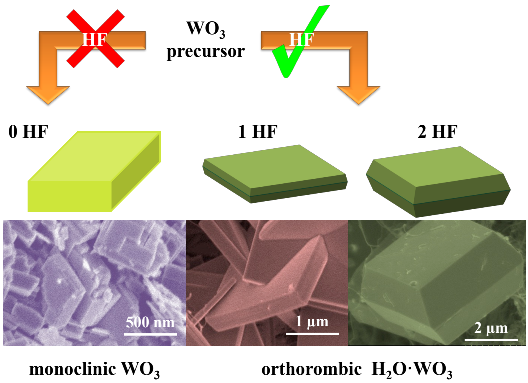

Figure 8. The effect of the concentration of the HF solution (crystallization time 1 h)

When HF was absent from the crystallization procedure, monoclinic WO_3 was obtained with nm sized crystals. When HF was present the formation of hydrates was favored with specific crystal geometry. When the concentration of HF was further raised, the development of new crystallographic planes was observed, while stabilizing the already appeared ones (*Figure 8*). Therefore, it was successfully shown, that HF can act as a shape-tailoring agent, which favors specific geometry and crystal phase composition.

Summary

The main aim of the research with TiO_2/WO_3 photocatalyst composites was to study the charge trapping ability of the WO_3 component in the composites. This was examined by changing the crystal structure and morphological properties of the WO_3 component.

For the TiO_2/WO_3/Au/MWCNT composites, it has been found that carbon nanotubes added at a sufficiently low concentration could have a positive effect on the photocatalytic activity of the composite. However, we cannot ignore the fact that both Au, Pt and the carbon nanotubes, and even the WO_3 component of the composite, can behave as electron acceptors, so they reduced rather than increased photocatalytic activity of the system.

After the successful selective photoreduction, the studies have shown that the localization of the noble metals within the TiO_2/WO_3 composites (on the surface of one of the oxides) was crucial. When the noble metal was located on TiO_2, the light absorption properties of the composites does not change significantly by increasing the concentration of WO_3 in TiO_2/WO_3/noble metal (Pt and Au) composites. However, otherwise (when the noble metal was located on WO_3) the light absorption properties were strongly dependent on the WO_3 content of the composites, while the photocatalytic activity of the composite decreases by increasing the concentration of WO_3 in TiO_2/WO_3/noble metal (Pt and Au) composites above a specific level (24 wt.%). It was found that the optimal WO_3 concentration could be within 10 wt.% in the case of the TiO_2/WO_3/noble metal (Pt and Au) composites.

The TiO_2/WO_3/Au composites produced by impregnation contained a low (4 wt.%) amount of WO_3, while tungsten was identified as a possible doping element. Furthermore, at this WO_3 content values both amorphous and crystalline WO_3 was

identified. So maybe not the presence of the noble metal, but the amorphous nature of WO_3 was beneficial on the photocatalytic activity of these composites.

Obtaining the WO_3 by hydrothermal crystallization, the effect of WO_3 structural, and crystalline properties were investigated. Based on the results of the material structure studies and the measured photocatalytic activity, we obtained TiO_2/WO_3 composites with a very good photocatalytic activity, when the WO_3 component was found as tungsten hydrate ($WO_3 \cdot H_2O$) or partial tungsten hydrate ($H_{0,23}WO_3$ or $WO_3(H_2O)_{0,5}$). The presence of hydrogen in hydrates may induce more crystal defects in the given crystal structure. In addition, tungsten hydrates or partial tungsten hydrates have less structured crystal structure, opposite to the anhydrous WO_3 crystal structures. Therefore, the defects and the structure and morphology of WO_3 crystals contributed to the efficient trapping of the photogenerated electrons, delaying the reaction of the electrons with the O_2 molecules or inhibiting the recombination process.

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SCIENTIFIC ACTIVITY

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Publications related to the scientific topic of the dissertation

Zs. Pap, É. Karácsonyi, L. Baia, L. C. Pop, V. Danciu, K. Hernádi, K. Mogyorósi, and A. Dombi: *TiO_2/WO_3/Au/MWCNT composite materials for photocatalytic hydrogen production: Advantages and draw-backs*, Physica Status Solidi, B 249, No. 12, 2592-2595 (2012) / DOI 10.1002/pssb.201200095

I.F.: 1.61

Hivatkozások száma: 6

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Conferences as co-author:

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