Preparation, characterisation and evaluation of photocatalytic activity of titanium dioxide based multiwalled carbon nanotube containing composites

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1. Introduction and Objectives

After the Industrial Revolution in the 18th-19th century, but mainly after the World War II humanity more and more took possession of the resources of the Earth. At that time people did not think of their environment; since then, environmental pollution became one of the most urging environmental issues. The question of water pollution is especially important because clean water is essential for every living being. 10 % of the world's population do not have access to clean drinking water nowadays. According to the recent report of Word Economic Forum the water crisis is the most serious problem regarding the society. Beside the wastewater treatment methods of proof there is need for advanced techniques which can remove pollutants resistant to conventional methods. The advantage of advanced oxidation processes (AOP) is the high cleaning efficiency which is due to the high reactivity of the produced oxidative radicals. Heterogeneous photocatalysis is a rapidly developing branch of AOP technologies. The principle of photocatalysis is the formation of oxidative radicals on the surface of semiconductor (nano)particles upon irradiation, which eventually leads to the non-selective degradation of pollutants.

The most widely used and most promising semiconductor metal oxide is titanium dioxide (TiO_2). The advantage of this material lies in its availability, low cost, non-toxicity, biocompatibility and physical/chemical/photochemical stability. However, the bandgap energy of the photocatalytically more active anatase phase is quite high (E_g(anatase) = 3.2 eV ≈ λ = 388 nm). Therefore, if one wants to use sunshine to promote photocatalytic reactions there are two possibilities: preparing photocatalysts which are active in visible irradiation or enhancing the already existing activity in UV irradiation.

Since the discovery of carbon nanotubes (CNT) in 1991 they are subjects of intense research due to their exceptional stability, mechanical and electric properties. These features make CNTs an excellent composite forming material. Taking advantage of the electric and adsorption properties of CNTs, TiO_2/MWCNT composites can be prepared showing elevated photocatalytic activity. Despite of the extensive research there is still no definite explanation regarding this activity-enhancing effect, but several theories already exist. According to these theories CNTs can either act as electron "sinks" and conductive channels for the photogenerated electrons thus reducing e^-/h^+ recombination rate, or CNTs can act as a photosensitizer.

Carbon nanotubes and related nanostructures are subjects of intense research in our research group since 1995. One application area of these materials is their incorporation into composites thus metal oxide/CNT composites were already investigated by our group. Other applications concern the photocatalytic behaviour of these metal oxide/CNT composites. During my doctoral work, I investigated the preparation and photocatalytic activity of TiO_2/MWCNT composite materials.

The aim of my PhD work was to study the different possibilities of preparation of TiO_2/MWCNT composites. I planned to investigate the effect of morphology on the photocatalytic activity of the composite samples prepared by sol-gel method. Later, based on preliminary findings I planned to refine the preparation method which provided photocatalysts with higher photocatalytic activity in order to perform controlled synthesis of TiO_2/MWCNT composites. I planned to study the effect of MWCNT content on the photocatalytic activity in case of two model compounds which have distinct chemical characteristics (phenol and oxalic acid).

I planned to investigate preparation methods which can result TiO_2/MWCNT composites containing anatase and rutile polymorphs with different ratios. Based on the literature two facile methods were chosen: crystallisation via heat treatment and hydrothermal process. I wished to study the forming crystal structure, phase composition of TiO_2 polymorphs and their morphology. I planned to investigate the effect of the above-mentioned parameters on the photocatalytic activity of TiO_2/MWCNT composites.

2. Experimental methods, procedures

Chemicals were used as received without further purification. MWCNTs were synthetised in Prof. László Forró's group in École Polytechnique Fédérale de Lausanne (Switzerland, Lausanne) over Fe-Co/$CaCO_3$ catalyst via CCVD method. Acetylene and nitrogen was used as carbon source and carrier gas, respectively. The product was purified by washing with diluted hydrochloric acid and functionalised by concentrated nitric acidic treatment.

By applying different titanium precursors, solvents and hydrolysis times I have prepared TiO_2/MWCNT composites of different morphology. These composites showed dissimilar photocatalytic activities. I have modified and refined the preparation methods, and synthesised new materials to investigate the effect of MWCNT content on the photocatalytic activity of TiO_2/MWCNT composites in photodegradation reactions of phenol and oxalic acid.

I have investigated preparation methods which are suitable for the synthesis of TiO_2/MWCNT composites with different anatase and rutile content. Firstly, I have prepared TiO_2/MWCNT composites with heat treatment. I have characterised the samples heat treated at different temperatures and determined their photocatalytic activities. After these experiments, another preparation method was studied. Hydrothermal crystallisation is a convenient method for preparation of shape- and phase-controlled nanomaterials. Acid concentration was varied during the hydrothermal process. I have characterised the morphology, phase composition and determined the photocatalytic activity of the prepared samples.

Crystal structure of the samples were characterised by powder X-ray diffraction (XRD) technique. A Rigaku Miniflex II instrument equipped with copper $K_{\alpha 1}$ (λ = 0.15418 nm) radiation source was used. By analysing the diffraction patterns information have been extracted about the crystallinity, phase composition and particle size.

Transmission electron microscopy (TEM) method was used to obtain information about the morphology of the samples. Philips CM10 (100 kV) and FEI Technai G^2 20 X-TWIN (200 kV) instruments were used. First, the sample was suspended in ethanol then dripped onto a copper TEM grid and dried before placing it into the instrument.

Thermogravimetric (TG) analysis was used to gain information about the thermal behaviour of the samples. This method was applied in order to study the thermal stability of MWCNTs and determine the MWCNT content of the samples. Netzsch STA 409 PC instrument was used in the described experiments.

Raman spectroscopy was used to determine the quality of MWCNTs and to prove the presence of MWCNTs in the composite materials. Furthermore, the interaction between the MWCNTs and the TiO_2 particles was also investigated. For Raman measurements a Thermo Scientific DXR Raman microscope with laser light source (λ = 562 nm) was used.

The specific surface areas of the samples were determined by nitrogen adsorption. A BEL Japan BELCAT-A sorption analyser was used to measure the nitrogen adsorption of samples.

Light absorption of solid samples was measured by an Avantes AvaSpec-ULS2048 UV-Visible fibre optic diode array spectrophotometer using $BaSO_4$ as reference material.

The TiO_2/MWCNT composites were tested in photodegradation reactions of phenol, oxalic acid and salicylic acid. Fluorescent UV-A lamp (λ_{max} = ~350 nm) was used in every experiment. I have designed a new photoreactor system. The advantage of this new system is using a low powered irradiation source (6 W) and a gas inlet which stirs the suspension which contains the photocatalyst. The concentration of phenol and oxalic acid was followed by HPLC technique while the concentration of salicylic acid was determined by UV-Vis method.

3. Summary of new scientific results

T1. The effect of the morphology of TiO_2/MWCNT on their photocatalytic activity

1.1. I have successfully prepared TiO_2/MWCNT composites with different morphology by varying certain synthesis parameters (precursor, solvent, rate of hydrolysis). These composites were tested in photocatalytic degradation reactions. Based on these findings it was found that having frequently positioned small TiO_2 nanoparticles on the surface of MWCNTs is advantageous from the photocatalytic point of view.

1.2. I have developed a sol-gel preparation process of TiO_2/MWCNT composites which is suitable for obtaining TiO_2/MWCNT composites with same morphology regardless of the MWCNT content. The average diameter of the deposited TiO_2 nanoparticles on the surface of MWCNTs was found to be in the range of 10-14 nm. Each composite was characterised by various techniques resulting significant differences only in their light absorption properties.

1.3. I have demonstrated that if the MWCNTs and the TiO_2 form a physical mixture the overall photocatalytic activity decreases. Moreover, with the increase of TiO_2 particle size in the composite sample (roughly above 50 nm), the photocatalytic activity decreases likewise.

T2. The effect of MWCNT content on the photocatalytic activity of TiO_2/MWCNT composites

2.1. TiO_2/MWCNT composites prepared with different MWCNT content were tested in phenol and oxalic acid photodegradation reactions. I have determined that the photocatalytic activity follows a maximum curve-like change with the increasing MWCNT content in case of both model compounds. Maximum photocatalytic efficiency was reached at 1 m/m% of MWCNT content in phenol, and 5 m/m% in oxalic acid photodegradation. The sample containing 10 m/m% MWCNT showed the weakest photocatalytic activity both in phenol and oxalic acid photodegradation reaction. This low photocatalytic activity can be explained with the different light absorption properties of the composite samples.

2.2. Photocatalytic performance of a certain sol-gel prepared TiO_2/MWCNT composite differs radically depending on the nature of the examined model compound (oxalic acid, phenol). The composites showed higher photocatalytic activity in case of oxalic acid. Based on our results and literature data, the reason behind this phenomenon was found to be the difference in adsorption properties of phenol and oxalic acid on TiO_2 surface and thus the photodegradation mechanism dissimilarities of the two model compounds.

T3. Preparation and photocatalytic activity of TiO_2/MWCNT composites with different anatase/rutile ratio

3.1. In order to control the anatase/rutile ratio in TiO_2/MWCNT composites I used heat treatment preparation method in inert atmosphere. The rutile content of the composite samples increased simultaneously with the increase of heat treatment temperature, which also caused drastic changes in the morphology. The TiO_2 particles were bound to the surface of MWCNTs in case of the sample heat treated at 400 °C. At higher temperatures, the TiO_2 particles started to sinter into bigger crystals; detachment of particles from the surface of MWCNTs was also observed. The photocatalytic activity of the samples decreased with increasing heat treatment temperature in salicylic acid photodegradation reaction. I have found that this decrease of photocatalytic efficiency can be attributed to three reasons: the detachment of TiO_2 particles from MWCNT surface, the increase of TiO_2 particle size and the formation of rutile phase.

3.2. I have successfully prepared TiO_2/MWCNT composites with different anatase/rutile ratio via combined sol-gel/hydrothermal process by varying the applied hydrochloric acid concentration during the synthesis procedure. The composites contained anatase phase TiO_2 with the presence of brookite under neutral and dilute acidic conditions. I have identified the acid concentration range ($1\cdot10^{-1}$ – 1 mol dm^{-3}) where the anatase and the brookite phase transforms into rutile phase TiO_2. I have established a phase transition order: (a,b) → (a,b,r) → (b,r) → (r) where 'a', 'b' and 'r' refers to anatase, brookite and rutile, respectively. This phase transition order is in good agreement with the one of pure TiO_2 material found in literature in absence of MWCNTs. The morphology of samples can be considered uniform from the neutral to 0.3 mol dm^{-3} acid concentration; TiO_2 nanoparticles of 15-25 nm in diameter were located on the surface of MWCNTs as well as between them.

3.3. I have also analysed the photocatalytic activity of TiO_2/MWCNT composites synthesised via sol-gel/hydrothermal process. The composites prepared under neutral and dilute acidic conditions (containing brookite beside anatase) showed significant photocatalytic activity. The TiO_2/MWCNT composite crystallised in 0.3 mol dm^{-3} hydrochloric acid showed the highest photocatalytic activity among the composites prepared by sol-gel/hydrothermal process. This material composed of 11 m/m% rutile, 41 m/m% anatase and 48 m/m% brookite phase. According to my findings, small amount of rutile can enhance the photocatalytic activity of TiO_2/MWCNT composites. Further increase of the rutile content influences negatively the photocatalytic activity of the composites.

4. Scientific publication

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ΣΣIF = 35.417

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