

# Heterogeneous Photocatalysis: A Solution for a Greener Earth

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Since the beginning of the industrial era, various human activities have steadily increased, leading to rapid technological developments and high population growth. In consequence, the expanding industry has heavily polluted the atmosphere, soil, and water, with negative consequences for humans and the environment.

To decrease pollution emissions, various chemical, physical, and biological treatment methods have been developed. The major technics for treating wastewater are based on wastewater treatment plants using dry cleaning, decantation, and biological treatments. Occasionally, pollutant molecules are not eliminated by these processes; therefore, other technics can be used as secondary treatments to remove these small residual fractions of pollution. Among these methods, photocatalysis is a well-developed technic in the past few years. Through a photocatalyst and using light's energy, photocatalysis allows the production of highly reactive species that can react and decompose organic molecules, yielding, in the best case, the final decomposition products CO<sub>2</sub> and H<sub>2</sub>O. The most commonly used photocatalysts are titanium dioxide (TiO<sub>2</sub>), zinc oxide (ZnO), and tin oxide (SnO<sub>2</sub>).

The papers contributing to this Special Issue address innovative photocatalytic processes for environmental applications. In what follows, we provide a synopsis of the obtained results in the 17 papers published in this Special Issue.

## 1. TiO<sub>2</sub>-based photocatalysts

In this collection of articles, TiO<sub>2</sub> was used as the photocatalyst for depollution applications, and different doping and shaping were used to produce photoefficient materials.

In a contribution by Almeida et al. [1], the efficiency of photodegradation, the volatilization profile of bidentates, and the role of reactive oxidizing species (ROS) were explored for nanocrystalline TiO<sub>2</sub> modified with bidentate ligands (acetylacetonate). In this study, TiO<sub>2</sub>-ACAC CTC calcined at 300 °C (TiO<sub>2</sub>-A300) was applied for the photocatalytic degradation of chlorophenol (4-CP) and tetracycline (TC) under low power visible light (26 W). Furthermore, the ROS scavengers isopropanol and benzoquinone were added for studying the photocatalytic role of •OH and •O<sub>2</sub><sup>-</sup> radicals. The photocatalytic abatement of tetracycline (68.6%), performed via TiO<sub>2</sub>-A300, was two times higher than that observed for chlorophenol (31.3%) after 6 h, indicating the distinct participation of ROS in the degradation of these pollutants. The addition of the ROS scavenger revealed •O<sub>2</sub><sup>-</sup> radicals as primarily responsible for the high efficiency of TiO<sub>2</sub>-ACAC CTC under reduced visible light. On the other hand, the •OH radicals were not efficiently generated in the CTC. Therefore, the development of heterostructures based on TiO<sub>2</sub>-ACAC CTC can increase the generation of ROS through coupling with semiconductors capable of generating •OH under visible light.

In the study of Pascariu et al. [2], Ag–TiO<sub>2</sub> nanostructures were prepared via electrospinning, followed by calcination at 400 °C. Morphological characterization revealed the presence of one-dimensional uniform Ag–TiO<sub>2</sub> nanostructured nanofibers, with a diameter from 65 to 100 nm, depending on the Ag loading, composed of small crystals interconnected



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with each other. Structural characterization indicated that Ag was successfully integrated as small nanocrystals without affecting much of the TiO<sub>2</sub> crystal lattice. Moreover, the presence of nano-Ag was found to contribute to reducing the band gap energy, which enables the activation by the absorption of visible light while, at the same time, delaying the electron–hole recombination. Tests of their photocatalytic activity in methylene blue, amaranth, Congo red, and orange II degradation revealed an increase by more than 20% in color removal efficiency at an almost double rate for the case of 0.1% Ag–TiO<sub>2</sub> nanofibers compared with pure TiO<sub>2</sub>.

In another study [3], Mancuso et al. successfully prepared different tri-doped TiO<sub>2</sub> photocatalysts (Fe-N-P/TiO<sub>2</sub>, Fe-N-S/TiO<sub>2</sub>, Fe-Pr-N/TiO<sub>2</sub>, Pr-N-S/TiO<sub>2</sub>, and P-N-S/TiO<sub>2</sub>) and tested them in the photocatalytic removal of thiacloprid (THI) under UV-A, visible, and direct solar light irradiation. The physical–chemical properties of the prepared catalysts revealed that dopants were effectively incorporated into the anatase TiO<sub>2</sub> lattice, resulting in a decrease in the energy band gap. The reduction in photoluminescence intensity indicated a lower combination rate and longer lifespan of photogenerated carriers of all doped samples in comparison with the undoped TiO<sub>2</sub>. The doped photocatalysts promoted photodegradation under UV-A light irradiation and also extended the optical response of TiO<sub>2</sub> to the visible light region. Fe-N-P tri-doped TiO<sub>2</sub> sample exhibited the highest THI photodegradation degree (64% under UV-A light, 29% under visible light, and 73% under solar light).

In the contribution of Benkhenouche-Bouchene et al. [4], TiO<sub>2</sub> that was prepared using a green aqueous sol–gel peptization process was co-doped with nitrogen and zirconium to improve and extend its photoactivity to the visible region. For all doped and co-doped samples, TiO<sub>2</sub> nanoparticles with sizes ranging from 4 to 8 nm were formed of anatase–brookite phases. X-ray photoelectron (XPS) measurements showed that nitrogen was incorporated into the TiO<sub>2</sub> materials through Ti–O–N bonds, allowing light absorption in the visible region. The XPS spectra of the Zr-(co)doped powders showed the presence of TiO<sub>2</sub>–ZrO<sub>2</sub> mixed oxide materials. Under visible light, the best co-doped sample yielded a degradation of p-nitrophenol (PNP) equal to 70%, instead of 25% with pure TiO<sub>2</sub> and 10% with P25 under the same conditions. Similarly, the photocatalytic activity improved under UV–Vis, reaching 95% with the best sample, compared with 50% with pure TiO<sub>2</sub>.

In the study of Eun et al. [5], sol–gel-synthesized N-doped and carbon–nitrogen–sulfur (CNS)-doped TiO<sub>2</sub> solutions were deposited on upconversion phosphor using a dip-coating method. Scanning electron microscopy (SEM) imaging showed that there was a change in the morphology of TiO<sub>2</sub> coated on NaYF<sub>4</sub>:Yb,Er from spherical to nanorods caused by additional urea and thiourea doping reagents. Fourier transform infrared (FTIR) spectroscopy further verified the existence of nitrate–hyponitrite, carboxylate, and SO<sub>4</sub><sup>2−</sup> because of the doping effect. NaYF<sub>4</sub>:Yb,Er composites coated with N- and CNS-doped TiO<sub>2</sub> exhibited a slight shift in UV–Vis spectra toward the visible light region. The photocatalytic reactivity with CNS-doped TiO<sub>2</sub>/NaYF<sub>4</sub>:Yb,Er surpassed that of the undoped TiO<sub>2</sub>/NaYF<sub>4</sub>:Yb,Er for the MB solution and toluene. The photocatalytic activity was increased by CNS doping of TiO<sub>2</sub>, which improved light sensitization as a result of band gap narrowing due to impurity sites.

In the presented study by Mahy et al. [6], the objective was to improve the efficiency of TiO<sub>2</sub> photocatalysts via activation treatments and through modification with palladium nanoparticles and doping with SiO<sub>2</sub>. X-ray diffraction provided evidence that the crystallographic structure of TiO<sub>2</sub> was anatase and that Pd was present, either in its oxidized form after calcination or in its reduced form after reduction. The results on methylene blue degradation showed that the photocatalytic activity of the catalysts was inversely proportional to the content of silica present in the matrix. A small amount of silica improved the photocatalytic activity, compared with the pure TiO<sub>2</sub> sample. By contrast, a high amount of silica delayed the crystallization of TiO<sub>2</sub> in its anatase form. The introduction of Pd species increased the photocatalytic activity of the samples because it allowed for a decrease in the rate of electron–hole recombination in TiO<sub>2</sub>. The reduction treatment improved the activity

of photocatalysts, regardless of the palladium content, owing to the reduction of  $Ti^{4+}$  to  $Ti^{3+}$ , and the formation of defects in the crystallographic structure of anatase.

A review of the green synthesis of  $TiO_2$  is also presented in this issue [7]. Indeed, in this study, the authors reported an eco-friendly process for producing  $TiO_2$  via colloidal aqueous sol-gel synthesis, resulting in crystalline materials without a calcination step. Three types of colloidal aqueous  $TiO_2$  were reviewed: the as-synthesized type obtained directly after synthesis, without any specific treatment; the calcined, obtained after a subsequent calcination step; and the hydrothermal, obtained after a specific autoclave treatment. This eco-friendly process is based on the hydrolysis of a Ti precursor in excess of water, followed by the peptization of the precipitated  $TiO_2$ . Depending on the synthesis parameters, the three crystalline phases of  $TiO_2$  (anatase, brookite, and rutile) can be obtained. The morphology of the nanoparticles can also be tailored by the synthesis parameters. The most important parameter is the peptizing agent. Indeed, depending on its acidic or basic character and also on its amount, it can modulate the crystallinity and morphology of  $TiO_2$ . Colloidal aqueous  $TiO_2$  photocatalysts are mainly being used in various photocatalytic reactions for organic pollutant degradation. The as-synthesized materials seem to have equivalent photocatalytic efficiency to the photocatalysts post-treated with thermal treatments and the commercial Evonik Aeroxide P25, which is produced via a high-temperature process.

$TiO_2$  in form of film is also presented in the contribution of De Ceglie et al. [8]. In this study, the photocatalytic efficiency of an innovative UV-light catalyst consisting of a mesoporous  $TiO_2$  coating on glass fibers was investigated. Photocatalytic activity of the synthesized material was tested, for the first time, on a secondary wastewater effluent spiked with nine pharmaceuticals (PhACs), and the results were compared with the photolysis used as a benchmark treatment. Interestingly, the novel photocatalyst led to an increase in the degradation of carbamazepine and trimethoprim (about 2.2 times faster than the photolysis). Several transformation products (TPs) resulting from both the spiked PhACs and the compounds naturally occurring in the secondary wastewater effluent were identified through UPLC-QTOF/MS/MS. Some of them, produced mainly from carbamazepine and trimethoprim, were still present at the end of the photolytic treatment, while they were completely or partially removed by the photocatalytic treatment.

## 2. Composite materials as photocatalysts

In the papers under this theme, different composite materials were produced to be used as photocatalysts.

In one study [9], Ghazzy et al. reported a visible-light-induced, trimetallic catalyst ( $Cu_{0.5}Zn_{0.5}Fe_2O_4$ ) prepared through green synthesis using Tilia plant extract. The spinel crystalline material was ~34 nm. In benign reaction conditions, the prepared photocatalyst oxidized various benzylic alcohols with excellent yield and selectivity toward aldehyde with 99% and 98%, respectively. Aromatic and aliphatic alcohols (such as furfuryl alcohol and 1-octanol) were photocatalytically oxidized using  $Cu_{0.5}Zn_{0.5}Fe_2O_4$ , LED light, and  $H_2O_2$  as oxidant, 2 h reaction time, and ambient temperature. The advantages of the catalyst were found in terms of reduced catalyst loading, activating catalyst using visible light in mild conditions, high conversion of the starting material, and recyclability of up to 5 times without loss of selectivity.

In the contribution of Mahy et al. [10], raw clays, from Cameroon, were modified with semiconductors ( $TiO_2$  and  $ZnO$ ) to improve their depollution properties with the addition of photocatalytic properties.  $Cu^{2+}$  ions were also added to the clay via an ionic exchange to increase the specific surface area. The presence of  $TiO_2$  and  $ZnO$  was confirmed by the detection of anatase and wurtzite, respectively. The composite clays showed increased specific surface areas. The adsorption property of the raw clays was evaluated on two pollutants—namely, fluorescein (FL) and p-nitrophenol (PNP). The experiments showed that the raw clays can adsorb FL but were not efficient for PNP. To demonstrate the photocatalytic property resulting from the added semiconductors, photocatalytic experiments were performed under UV-A light on PNP. These experiments showed degradation up to

90% after 8 h of exposure with the best ZnO-modified clay. The proposed treatment of raw clays seems promising to treat pollutants, especially in developing countries.

In another contribution [11], Dao et al. prepared co-doped NiTiO<sub>3</sub>/g-C<sub>3</sub>N<sub>4</sub> composite photocatalysts using a modified Pechini method to improve their photocatalytic activity toward methylene blue photodegradation under visible light irradiation. The combination of Co-doped NiTiO<sub>3</sub> and g-C<sub>3</sub>N<sub>4</sub> and Co-doping into the NiTiO<sub>3</sub> lattice synergistically enhanced the photocatalytic performance of the composite photocatalysts. X-ray photoelectron spectroscopy results for the Co-doped NiTiO<sub>3</sub>/g-C<sub>3</sub>N<sub>4</sub> composite confirmed Ti–N linkages between the Co-doped NiTiO<sub>3</sub> and g-C<sub>3</sub>N<sub>4</sub>. In addition, characteristic X-ray diffraction peaks for the NiTiO<sub>3</sub> lattice structure clearly indicated the substitution of Co into the Ni–TiO<sub>3</sub> lattice structure. The composite structure and Co-doping of the C–x composite photocatalysts (x wt % Co-doped NiTiO<sub>3</sub>/g-C<sub>3</sub>N<sub>4</sub>) decreased the emission intensity of the photoluminescence spectra but also the semicircle radius of the Nyquist plot in electrochemical impedance spectroscopy, giving the highest K<sub>app</sub> value ( $7.15 \times 10^{-3} \text{ min}^{-1}$ ) for the Co-1 composite photocatalyst.

In the study of Bazta et al. [12], pure and Ce-modified ZnO nanosheet-like polycrystalline samples were successfully synthesized via a microwave-based process. The XRD results showed that the obtained photocatalysts were composed of hexagonal, wurtzite-type crystallites in the 34–44 nm size range. The microscopy showed nanosheet-shaped crystallites, with a composition close to Ce<sub>0.68</sub>Zn<sub>0.32</sub>O<sub>x</sub>. Importantly, the STEM–XEDS characterization of the photocatalyst samples revealed that Ce did not incorporate into the ZnO crystal lattice as a dopant but that a heterojunction formed between the ZnO nanosheets and the Ce–Zn mixed oxide phase nanoparticles. The optical analysis revealed that in the ZnO:Ce samples optical band gap was found to decrease to 3.17 eV in the samples with the highest Ce content. It was also found that the ZnO:Ce (2 at.%) sample exhibited the highest photocatalytic activity for the degradation of methylene blue (MB) when compared with both the pure ZnO and commercial TiO<sub>2</sub>–P25 under simulated sunlight irradiation.

### 3. Pilot and comparative process studies, and other photocatalytic processes

In this last collection of papers, pilot reactors were tested for larger-scale photocatalytic processes, and other advanced oxidative processes were explored.

In one study [13], Faccani et al. offered easily scalable solutions for adapting TiO<sub>2</sub>-based photocatalysts, which were deposited on different kinds of fabrics and implemented in a 6 L semipilot plant, using the photodegradation of rhodamine B (RhB) as a model of water pollution. They took advantage of a multivariable optimization approach to identify the best design options in terms of photodegradation efficiency and turnover frequency (TOF). Surprisingly, in the condition of use, the irradiation with a light-emitting diode (LED) visible lamp appeared as a valid alternative to the use of UV LED. The identification of the best design options in the semipilot plant allowed scaling up the technology in a 100 L pilot plant suitable for the treatment of industrial wastewater.

In the presented paper by Gonzalez-Burciaga et al. [14], several advanced oxidative processes were used to degrade 6-mercaptopurine (6-MP), a commonly used cytostatic agent. To degrade 6-MP, three processes were applied: photolysis (UV-C), photocatalysis (UV-C/TiO<sub>2</sub>), and their combination with H<sub>2</sub>O<sub>2</sub>. Each process was performed with variable initial pH (3.5, 7.0, and 9.5). Pilot-scale reactors were used, using UV-C lamps as a radiation source. Kinetic calculations for the first 20 min of reaction proved the significance of the addition of H<sub>2</sub>O<sub>2</sub>: In UV-C experiments, the highest k was reached under pH 3.5,  $k = 0.0094 \text{ min}^{-1}$ , while under UV-C/H<sub>2</sub>O<sub>2</sub>,  $k = 0.1071 \text{ min}^{-1}$  was reached under the same initial pH; similar behavior was observed for photocatalysis since k values of 0.0335 and  $0.1387 \text{ min}^{-1}$  were calculated for UV-C/TiO<sub>2</sub> and UV-C/TiO<sub>2</sub>/H<sub>2</sub>O<sub>2</sub> processes, respectively, also under acidic conditions. Degradation percentages here reported for UV-C/H<sub>2</sub>O<sub>2</sub> and UV-C/TiO<sub>2</sub>/H<sub>2</sub>O<sub>2</sub> processes were above 90% for all tested pH values.

In another contribution [15], Gonçalves et al. investigated the impact of different oxidation processes on the maprotiline degradation pathways via liquid chromatography–high-resolution mass spectrometry (LC/HRMS) experiments. Semiconductors photocatalysts—



namely, Fe–ZnO, Ce–ZnO, and TiO<sub>2</sub>—proved to be more efficient than heterogeneous photo-Fenton processes in the presence of hydrogen peroxide and persulfate. No significant differences were observed in the degradation pathways in the presence of photocatalysis, while the SO<sub>4</sub><sup>−</sup>-mediated process promoted the formation of different transformation products (TPs). Species resulting from ring openings were observed with higher persistence in the presence of SO<sub>4</sub><sup>−</sup>. In silico tests on mutagenicity, developmental/reproductive toxicity, Fathead minnow LC50, D. Magna LC50, and fish acute LC50 were carried out to estimate the toxicity of the identified transformation products. Low toxicant properties were estimated for TPs resulting from hydroxylation onto the bridge rather than aromatic rings, as well as those resulting from ring openings.

In the contribution of Tejera et al. [16], an integral treatment process for landfill leachate reverse osmosis concentrate (LLROC) was designed and assessed, aiming to reduce organic matter content and conductivity, as well as to increase its biodegradability. The process consisted of three steps. First, a coagulation/flocculation treatment (removal of the 76% chemical oxygen demand (COD), 57% specific ultraviolet absorption (SUVA), and 92% color) was utilized. In the second step, a photo-Fenton process resulted in enhanced biodegradability (i.e., the ratio between the biochemical oxygen demand (BOD<sub>5</sub>) and the COD increased from 0.06 to 0.4), and an extra 43% of the COD was removed at the best-trialed reaction conditions. A UV-A light-emitting diode lamp was tested and compared with conventional high-pressure mercury vapor lamps, achieving a 16% power consumption reduction. Finally, an optimized 30 g L<sup>−1</sup> lime treatment was implemented, which reduced conductivity by 43%, and the contents of sulfate, total nitrogen, chloride, and metals by 90%. Overall, the integral treatment of LLROC achieved the removal of 99.9% color, 90% COD, 90% sulfate, 90% nitrogen, 86% Al, 77% Zn, 84% Mn, 99% Mg, and 98% Si, in addition to significantly increasing biodegradability up to BOD<sub>5</sub>/COD = 0.4.

In the presented paper by Selvanathan et al. [17], the green synthesis of nickel oxide nanoparticles using phytochemicals from three different sources was employed to synthesize nickel oxide nanoparticles (NiO<sub>x</sub> NPs) as an efficient nanomaterial-based electrocatalyst for water splitting. Nickel (II) acetate tetrahydrate was reacted in presence of aloe vera leaves extract, papaya peel extract, and dragon fruit peel extract, respectively, and the physicochemical properties of the biosynthesized NPs were compared with sodium hydroxide (NaOH)-mediated NiO<sub>x</sub>. Based on the average particle size calculation from Scherrer's equation, using X-ray diffractograms and field-emission scanning electron microscope analysis revealed that all three biosynthesized NiO<sub>x</sub> NPs had smaller particle sizes than that synthesized using the base. Aloe-vera-mediated NiO<sub>x</sub> NPs exhibited the best electrocatalytic performance, with an overpotential of 413 mV at 10 mA cm<sup>−2</sup> and a Tafel slope of 95 mV dec<sup>−1</sup>. Electrochemical surface area (ECSA) measurement and electrochemical impedance spectroscopic analysis verified that the high surface area, efficient charge-transfer kinetics, and higher conductivity of aloe-vera-mediated NiO<sub>x</sub> NPs contribute to its low overpotential values.

In conclusion, this Special Issue entitled "Heterogeneous Photocatalysis: A Solution for a Greener Earth" gives an overview of the latest advances in the development of innovative photocatalytic processes. These studies pave the path for the development of innovative processes for a greener Earth.

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