

EFFECT OF TiO₂ PRECURSOR ON PHASE COMPOSITION AND PHOTOCATALYTIC DEGRADATION OF METHYLENE BLUE

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In this study, we synthesized two TiO₂ powders starting from two different titania precursors - titanium isopropoxide (TiO₂ IP) and titanium (triethanolaminato) isopropoxide (TiO₂ NH₂), using the sol-gel method. The photocatalysts were structurally characterized using various techniques - Fourier-transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), X-ray diffraction (XRD), Dynamic Light Scattering analysis (DLS), and Zeta potential analysis. The photocatalytic activity was tested on a methylene blue solution, varying the reaction conditions to determine the optimal operating parameters. Irradiation was carried out with a UV lamp, and the contact time was 180 minutes. It was found that the samples synthesized in this work exhibit a photocatalytic activity similar to that of the commercial TiO₂.

Keywords: titania, methylene blue, photocatalysis, emerging pollutants.

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1. Introduction

A major contributor to aquatic environmental contamination is industrial wastewater that poses a serious threat to both human health and the equilibrium of

ecological systems. The textile industry's wastewater is one of the most contaminated industrial effluents due to its high chemical oxygen demand and the presence of hazardous colorful substances [1]. Because synthetic dyes are difficult to biodegrade and cannot be eliminated by traditional wastewater treatment methods, they have a high degree of environmental persistence. Their great stability to water, temperature, light, and other chemicals like soaps and detergents accounts for their environmental persistence and long-term negative impact [2].

One of the most popular synthetic dyes is methylene blue, which is used as a colorant for textiles and paper. Additionally, methylene blue is utilized in medications, food, and cosmetics. When used in prescribed dosages, methylene blue has therapeutic benefits; nevertheless, uncontrolled methylene blue released into wastewater and untreated wastewater discharge into natural emissaries have detrimental impacts on both human health and flora [2]. According to the EU Directive, methylene blue can be classified as toxic to aquatic organisms, affecting both algal growth and the oxygenation rate of the water by inhibiting photosynthesis [3]. Photocatalysis is a simple wastewater treatment process that uses light radiation and semiconductors to break down emerging pollutants, including dyes [4].

Table 1. (Photo)catalytic degradation of dyes.

Author	Catalyst	Irradiation	Pollutant	Efficiency	Ref.
Jayamani et al.	Palladium nanoparticles	- (NaBH ₄)	Congo red Methylene blue	92% 95%	[7]
Acedo-Mendoza et al.	Silver-supported zinc oxide catalyst	UV lamp	Methylene blue Methyl orange	100% 100%	[8]
Lopez et al.	Magnetic zinc oxide nanorods	UV lamp	Methylene blue	97%	[9]
Prabha et al.	Zinc oxide nanoparticles	Hg lamp	Rose Bengal dye	> 80%	[10]
Phuruangrat et al.	Hexagonal ZnO nanoplates	UV lamp	Methylene blue	94.15%	[11]
Bibi et al.	NiO/ ZnO hybrid composites	Natural sunlight	Methylene blue	97.9%	[12]
Yasin et al.	TiO ₂ nanoparticles supported on PET nanofibres	Xenon lamp	Methylene blue	88%	[13]
Ria et al.	ZnO catalyst supported by montmorillonite	UV lamp	Methylene blue Methyl orange	99.35% 91.17%	[14]
Acedo-Mendoza et al.	Copper oxide supported on zinc oxide	UV lamp	Methylene blue Methyl orange	99% 99%	[15]
Momeni et al.	Ag-ZnO nanocomposites	UV lamp Solar light	Methylene blue	98%	[16]
Pava-Gomez et al.	Copper-doped TiO ₂ powder and film	Visible radiation	Methylene blue	42.35% (powder) 25.10 % (film)	[17]
Raj et al.	Zinc doped nickel oxide nanostructures	Sunlight	Rhodamine B 4-Nitrophenol	95% 80%	[18]

Among all semiconductor photocatalysts, TiO₂ is receiving the most attention due to its remarkable properties, which include chemical stability, high photocatalytic activity, the ability to be coated as a thin film on a substrate, nontoxicity, and environmental friendliness [5].

The following factors have an impact on photocatalytic processes:

- dye concentration: the catalyst must degrade a significant amount of dye. A small amount of dye, proportional to the initial dye concentration, is adsorbed on the catalyst surface. The initial dye concentration is inversely correlated with the percentage of dye degradation;

- amount of catalyst: in heterogeneous catalysis, increasing the amount of catalyst leads to an increase in the percentage of dye degradation due to the increase in the number of active sites. The amount of catalyst should not be particularly large because there is a risk that light radiation will no longer pass through the dye solution, due to aggregation of catalyst particles;

- pH: depending on the surface potential of the catalyst and the properties of the dye, the photocatalytic reaction can be induced or inhibited. By changing the pH of the solution, the surface potential of the catalyst can be modified, and thus the adsorption of pollutant molecules can be altered;

- irradiation time and light intensity: at high light intensities, the formation of excitons and the slow recombination of e^-h^+ pairs cause the photo-degradation percentage to be inversely correlated with light intensity [6].

The (photo)catalytic degradation of dyes is extensively documented in the specialized literature (Table 1).

The aim of the research is to synthesize TiO₂ powders, starting from two different titania precursors: titanium isopropoxide (TiO₂ IP) and titanium (triethanolaminate) isopropoxide (TiO₂ NH₂), via the sol-gel method. The samples were calcined at 700°C for 6 hours. The TiO₂ nanopowders were characterized by appropriate techniques. The photocatalytic activity was tested under various conditions - concentration of the methylene blue solution, amount of catalyst used, pH of the tested solution, and irradiation conditions to determine the optimal operating parameters. It is well known that precursors, synthesis conditions, and ternary components can tune the overall characteristics of the metal and metal-oxide particles, including photocatalytic activity. Considering this, the aim of this work is to compare the photocatalytic activity of the two different TiO₂ powders, obtained from the two different precursors which, to the best of our knowledge, have not been used previously to produce TiO₂, and to evaluate the photocatalytic activity.

2. Materials and Methods

2.1 Materials

Titanium isopropoxide, titanium (triethanolaminate) isopropoxide, and ethanol used for the synthesis were purchased from Sigma-Aldrich. Nitric acid 65% was purchased from Merck.

The following substances were used for testing the photocatalytic activity: methylene blue was produced by Merck; hydrochloric acid 1N and ammonia solution were purchased from Sigma-Aldrich. The commercial TiO_2 (anatase) sample was produced by Sigma-Aldrich.

2.2 Synthesis of TiO_2 powders

In a Berzelius beaker, 20 mL of titania precursor (titanium isopropoxide – TiO_2 IP or titanium (triethanolaminate) isopropoxide – TiO_2 NH_2) was added dropwise to 100 mL of ethanol, under constant stirring. After 2 hours, 15 mL of concentrated nitric acid and 60 mL of distilled water were added to the previously formed solution and kept at 60°C for 2 hours, under vigorous stirring, until the gel was obtained. The formed sol-gel was dried in a drying oven, initially at a temperature of 90 – 100°C , until the liquid was evaporated, and then at 300°C for 2 hours. The obtained product was ground in a mortar and subsequently calcined at 700°C for 6 hours (Figure 1).

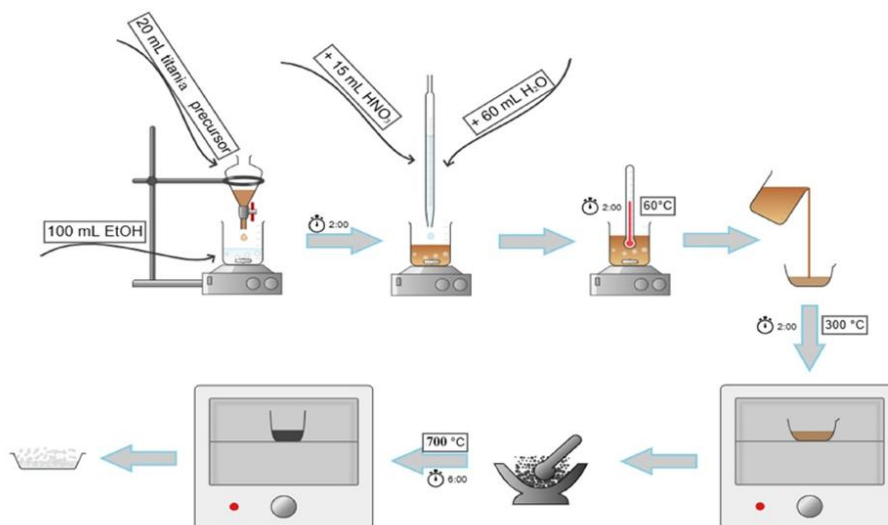


Figure 1. Synthesis of TiO_2 powders.

2.3 Characterization techniques

X-ray diffraction patterns (XRD) were recorded on a PANalytical X'Pert Pro MPD analyzer using $\text{Cu-K}\alpha$ radiation. Rietveld refinement was carried out

using a polynomial function for background approximation, a pseudo-Voigt function for peak profile, and a Caglioti function for peak width.

FTIR spectroscopy measurements were performed using a Thermo Nicolet iS50 FTIR spectroscope equipped with a diamond crystal-based ATR module capable of recording spectra over a range of 400 – 4000 cm⁻¹. To obtain a better signal-to-noise ratio, the spectra were obtained by summing up 32 scans recorded at a resolution of 4 cm⁻¹.

Scanning electron microscopy (SEM) images were recorded using a Quanta Inspect F - FEI microscope equipped with an EDS spectrometer, with the samples covered with a thin film of silver.

For DLS analysis, a DelsaMax Pro instrument (Beckman Coulter, Brea, CA, USA) was used. TiO₂ powder was dispersed in distilled water using an ultrasonic bath for 10 min to obtain a concentration of 1 mg/mL.

2.4 Photocatalytic activity

Photocatalytic activity was tested using a Herolab UV Hand Lamp ($\lambda=254$ nm). From previous tests, it was found that better results were obtained when using this wavelength, compared to the results obtained at the 365 nm wavelength. The irradiation time was 180 minutes, and the distance between the methylene blue solution and the lamp was set at 13.5 cm. The volume of methylene blue solution was 50 mL. To achieve the adsorption-desorption equilibrium, the TiO₂ and methylene blue suspension was kept in dark conditions, under stirring, for 30 minutes before starting irradiation. Every 30 minutes of irradiation, a volume of 5 mL was sampled and centrifuged at 5000 rpm for 5 minutes. The absorbance of the solution was measured at $\lambda=664$ nm, where methylene blue exhibits its absorption maximum. The percentage of methylene blue removal was calculated.

The photocatalytic activity was tested under various conditions: concentration of the methylene blue solution (3,2 ppm; 7 ppm; 10 ppm), amount of catalyst used (0.015 g; 0.030 g; 0.045 g; 0.060 g), pH of the tested solution (3; 6; 9), and irradiation conditions. We also tested a commercial TiO₂ (anatase) sample for comparison.

The lowest methylene blue concentration was selected since it was found in real textile industry effluents, according to data presented in the literature [19]. The highest concentration of methylene blue was chosen because some studies report on synthetic effluents, where the concentration is in the order of tens of mg/L [20].

3. Results and Discussions

3.1 XRD analysis

According to the XRD analysis (Figure 2), the TiO₂ IP sample is a mixture of the rutile and anatase crystalline forms, while for the TiO₂ NH₂ sample the signals corresponding to the rutile form were the majority, while the signals for the

anatase form were very weak. It was established that the commercial sample is a pure form of anatase. The anatase form was thus identified in the case of TiO_2 IP by the peaks corresponding to the crystalline planes (101), (004), (200), (105), (211), and (204), whereas the rutile form was identified by the peaks corresponding to the crystalline planes (110), (101), (200), (111), (210), and (211). The anatase-related signals in the TiO_2 NH_2 sample were generally quite weak, with the strongest peaks belonging to the (101) and (204) crystalline planes. In reference to the rutile form's crystalline planes, the signals that were found corresponded to the (110), (101), (200), (111), (210), and (211) planes.

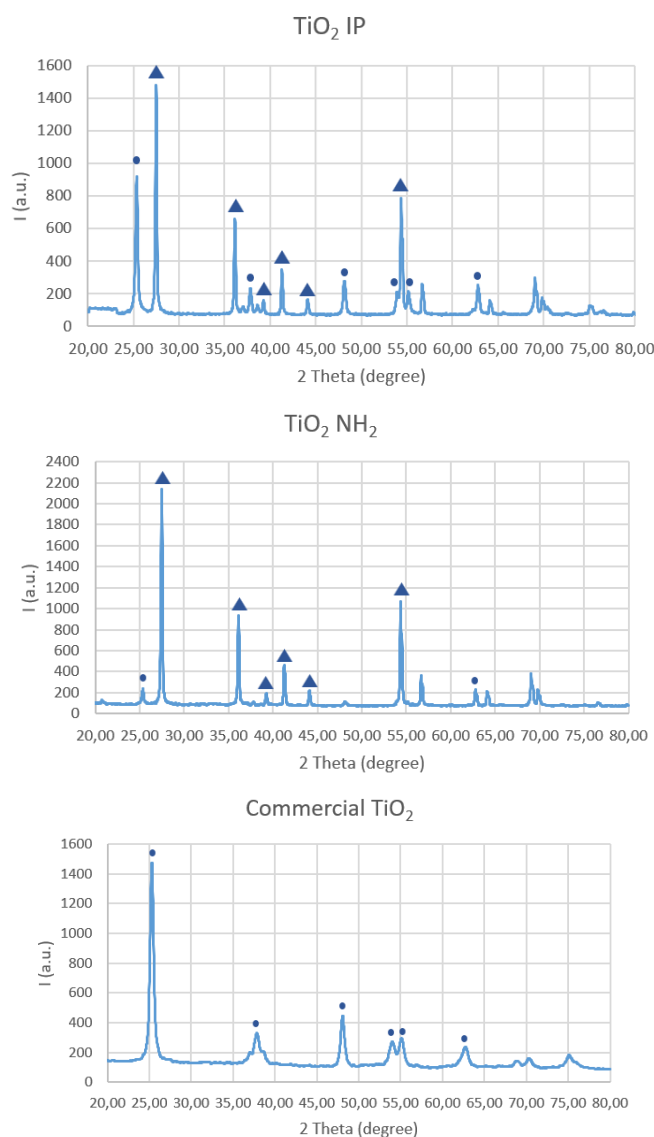


Figure 2. XRD patterns for TiO_2 IP, TiO_2 NH_2 and commercial TiO_2 (●=anatase; ▲=rutile)

3.2 FTIR analysis

The TiO₂ lattice vibration modes and the signals corresponding to the Ti-O and Ti-O-Ti bonds were identified between 400 and 800 cm⁻¹ in the FTIR spectra (Figure 3).

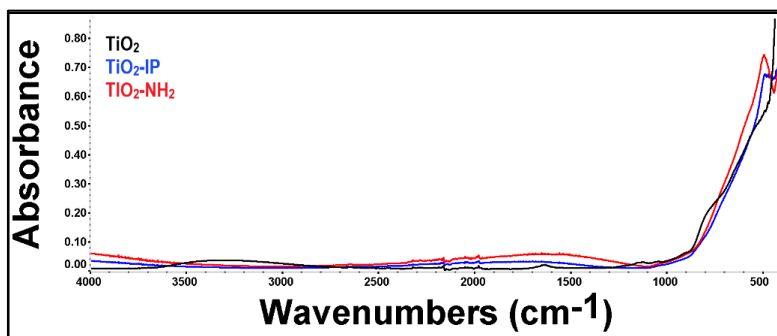
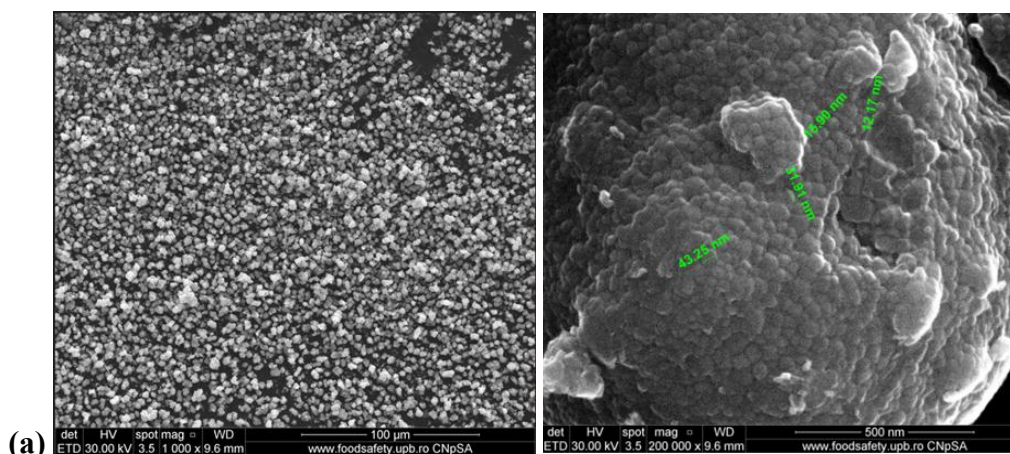


Figure 3. FTIR spectra for TiO₂ IP, TiO₂ NH₂ and commercial TiO₂

3.3 SEM analysis

The size and shape of the particles were determined by SEM analysis (Figure 4). The particles in the TiO₂ IP sample are aggregated and nearly spherical. The TiO₂ NH₂ sample's tetragonal prism particle morphology confirms the sample's high proportion of rutile form. Commercial TiO₂ particles have a similar appearance to TiO₂ IP.



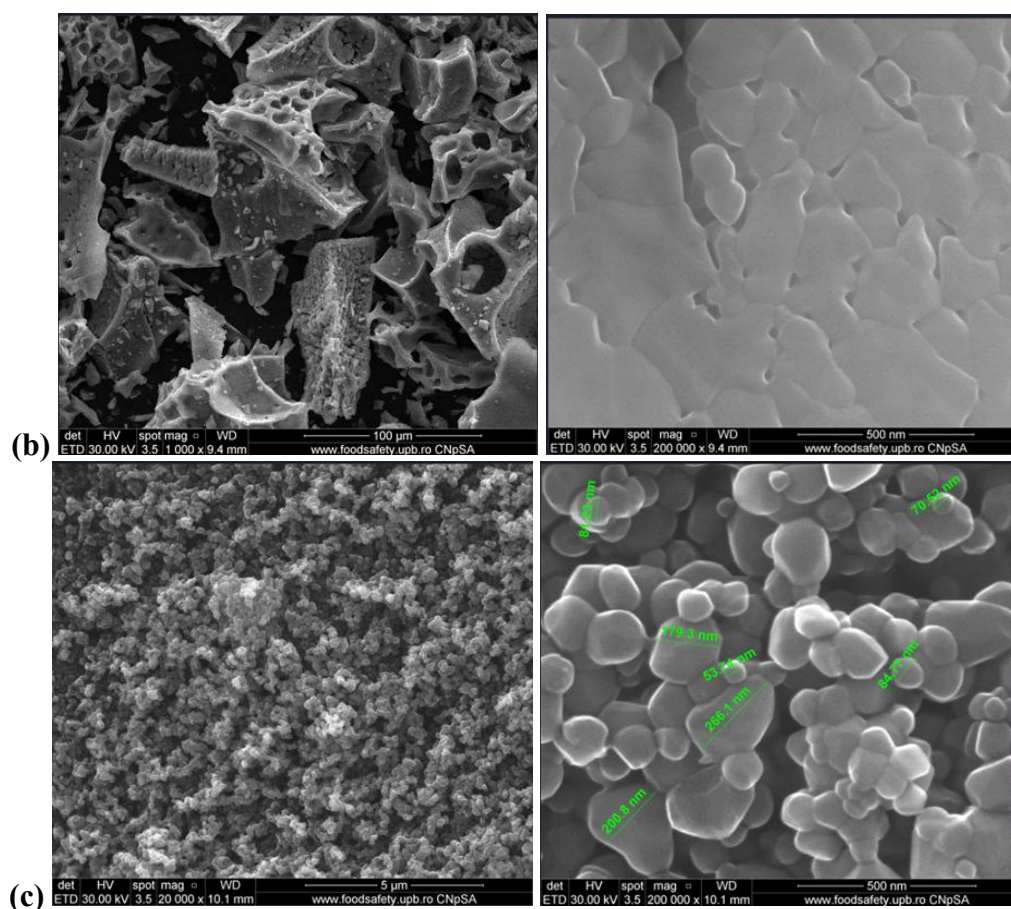


Figure 4. SEM images of TiO₂ IP (a), TiO₂ NH₂ (b) and commercial TiO₂ (c)

3.4 EDS analysis

The elements present in the commercial TiO₂ sample were identified and quantified using the EDS analysis. According to the EDS spectra (Figure 5), the sample contains 0.7% Al (weight percent), indicating that the TiO₂ particles were protected by a coating of Al₂O₃.

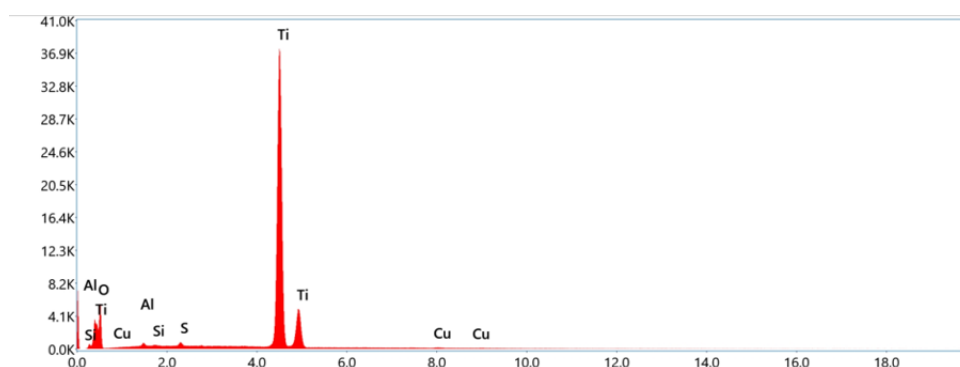


Figure 5. EDS spectra of commercial TiO₂

3.5 DLS analysis

One important indicator of colloidal dispersion stability is Zeta potential. Absolute values greater than 30 mV indicate a stable system and strong electrostatic repulsion between particles with similar electrical charge, preventing aggregation. In our case, the absolute values of the Zeta potentials of the synthesized samples are lower than 30 mV (Table 2), indicating that the particles can aggregate over time, which is also confirmed by the particle hydrodynamic diameter. The commercial TiO₂ sample has the lowest Zeta potential, suggesting an unstable dispersion and particles that tend to agglomerate quickly. As expected, TiO₂ NH₂ exhibited the strongest surface charge and the lowest hydrodynamic particles diameter. Considering the alumina coating, the commercial TiO₂ has a less pronounced tendency of agglomeration, and the hydrodynamic diameter seems to be comparable with the agglomerates observed in SEM (agglomerates of a few particles of 50-200nm). Overall, in the case of the commercial TiO₂ powder, the coalescence is minimal. In the case of the TiO₂ NH₂, based on the DLS it seems that these particles are the smallest but, combining with the SEM images it rather can conclude that in DLS only the fine sort of the particles remain in the suspension (with an average diameter of 437.1 ± 20.5 nm) while the largest particles (larger than tens of microns) are deposited from the suspension. In the case of TiO₂ IP, the diameter is 1203.8 ± 223.4 nm which is in good concordance with the SEM images, which means that the micrometric particles remain independent in aqueous solution (perhaps because of the $\sim -14.36 \pm 1.11$ mV zeta potential).

Table 2. Zeta potential and hydrodynamic particles diameter of commercial TiO₂, TiO₂ IP and TiO₂ NH₂.

Sample	Zeta Potential (mV)	Diameter (nm)
TiO ₂ IP	-14.36 ± 1.11	1203.8 ± 223.4
TiO ₂ NH ₂	-18.73 ± 2.44	437.1 ± 20.5
Commercial TiO ₂	-0.32 ± 0.06	759.2 ± 33.3

3.6 Photocatalytic activity

First, experiments were carried out to determine the ideal catalyst quantity for catalytic activity testing. A 3.2 ppm methylene blue solution was utilized for these experiments (Figure 6).

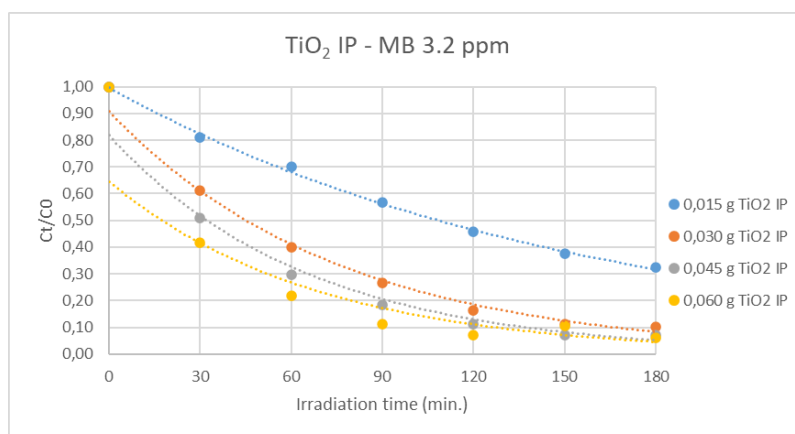
Similar degradation rates were achieved with 0.045 g and 0.060 g TiO_2 IP, respectively, yielding the highest percentages of degradation. Therefore, all the experiments were done with the smallest amount of catalyst, namely 0.045 g of TiO_2 IP.

The next step was to determine the influence of the concentration of the methylene blue solution on the photocatalytic efficiency. Three solutions were evaluated at 3.2 ppm, 7 ppm, and 10 ppm concentrations. As anticipated, the degradation percentage decreased as the concentration of the methylene blue solution increased; however, all dye solutions had degradation percentage values above 50%.

The impact of the solution pH on degradation efficiency was also examined using the highest concentration of methylene blue solution (10 ppm). Degradation efficiency was poor at acidic pH values, while neutral pH (pH=6) produced the greatest results, with values comparable to those at basic pH values.

In conclusion, when 0.045 g of TiO_2 IP was used to degrade a solution with a concentration of 3.2 ppm at neutral or basic pH values, the best results were obtained.

A similar procedure was followed for the TiO_2 NH_2 sample (Figure 7). In this case, the amount of catalyst for which the highest degradation percentage and the highest degradation rate were obtained was 0.060 g. The efficiency of the samples containing 0.030 g TiO_2 NH_2 and 0.045 g TiO_2 NH_2 were comparable. The next tests were conducted using 0.060 g TiO_2 NH_2 to investigate the effect of pH and dye solution concentration on degradation percentage.



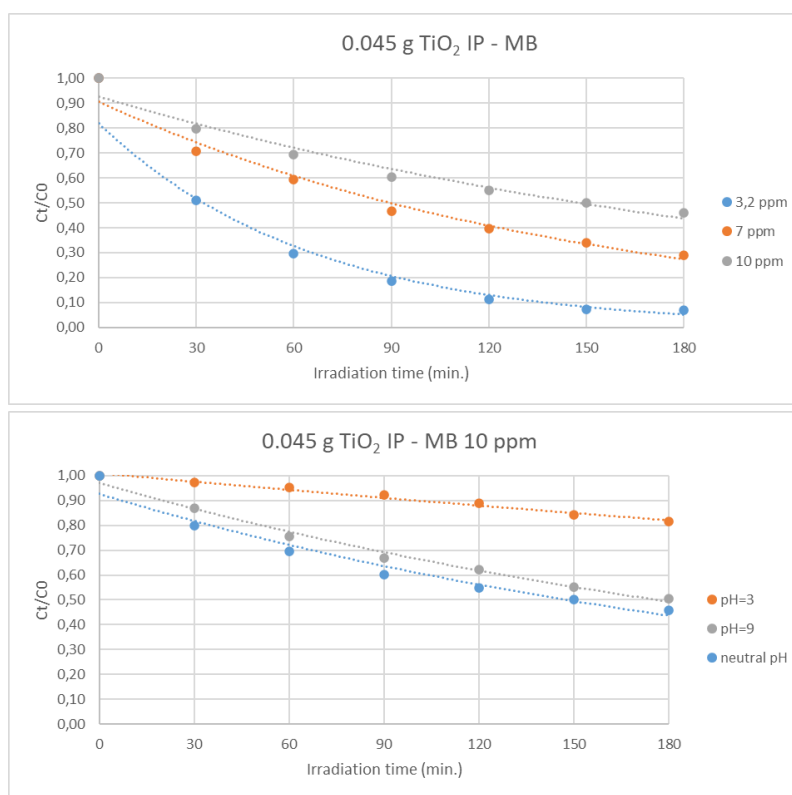
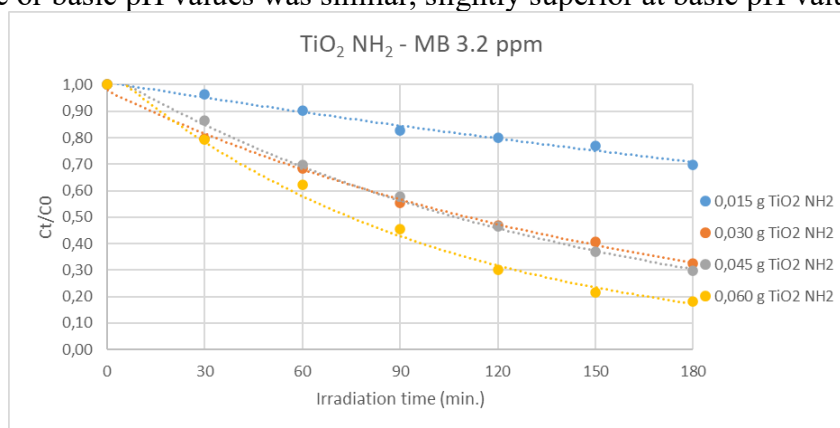


Figure 6. Photocatalytic activity of TiO₂ IP.

The tests conducted at various concentrations of methylene blue solution and 0.060 g TiO₂ NH₂ produced findings comparable to those of TiO₂ IP; however, the percentages and maximum degradation rates were lower in the case of TiO₂ NH₂. In terms of pH's influence, the results demonstrated that neutral pH values produced the highest degradation efficiency. The degradation percentage obtained at acidic or basic pH values was similar, slightly superior at basic pH values.



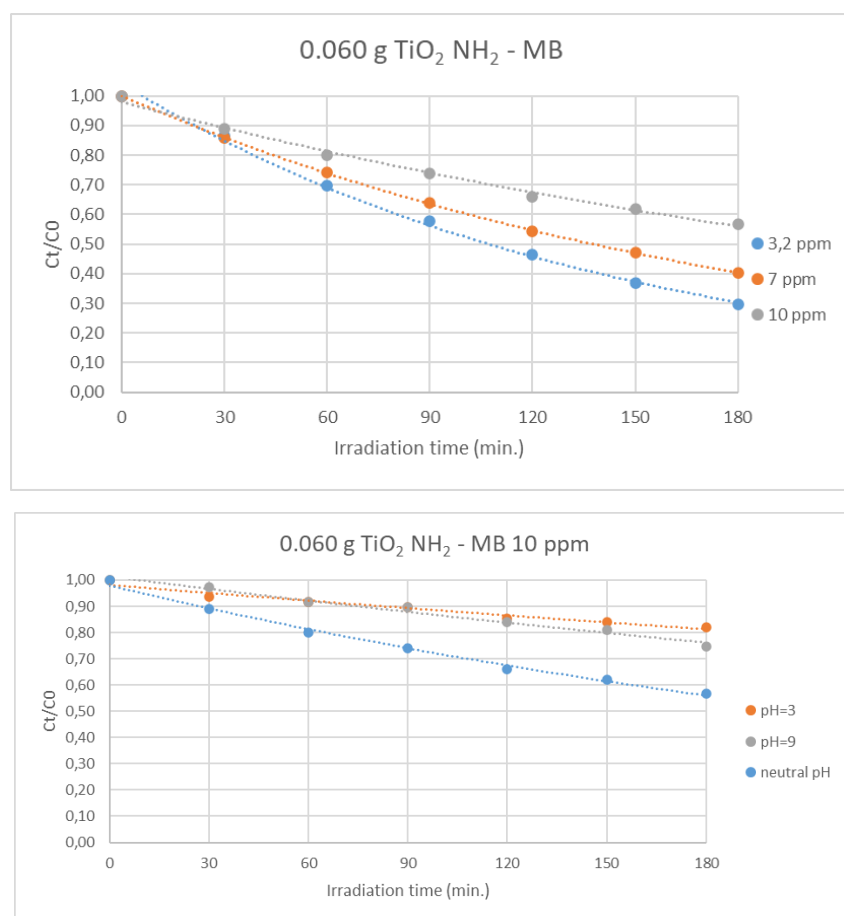
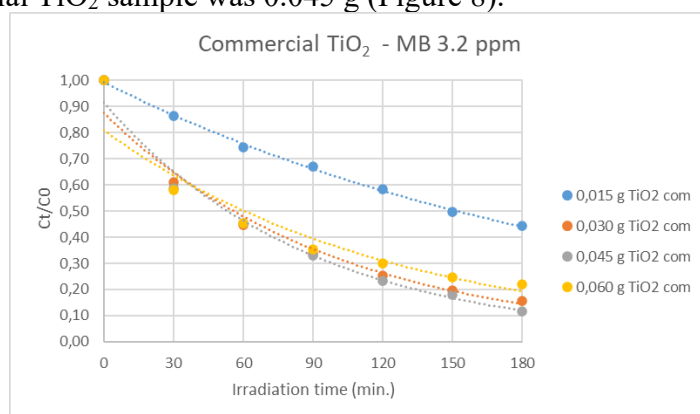


Figure 7. Photocatalytic activity of TiO₂ NH₂.

The amount of catalyst that produced the highest efficiency in the case of the commercial TiO₂ sample was 0.045 g (Figure 8).



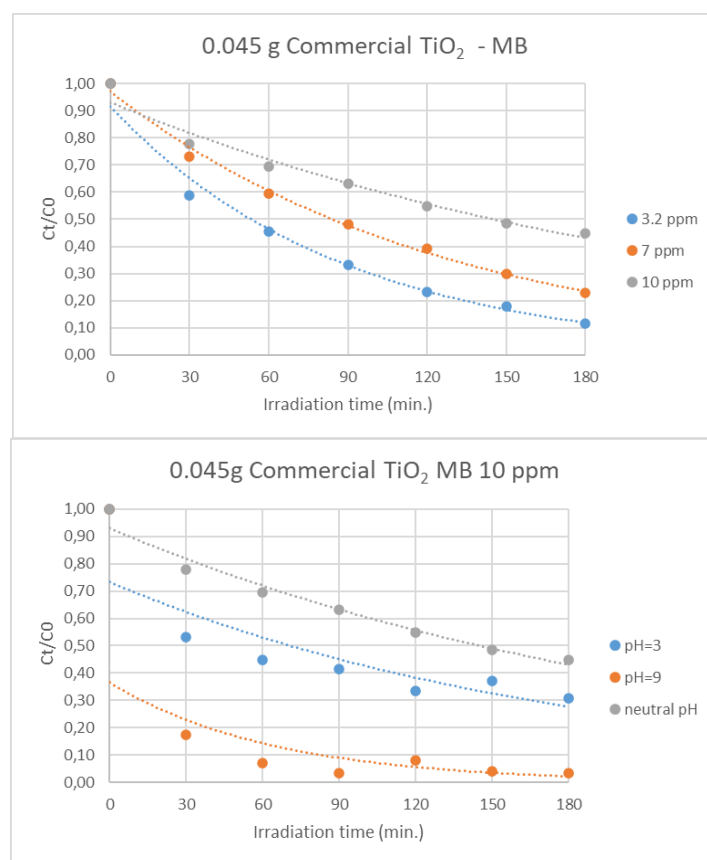


Figure 8. Photocatalytic activity of commercial TiO_2 .

When 0.060 g of commercial TiO_2 was used, the degradation efficiency was lower than in samples with 0.030 g and 0.045 g of catalyst. This was most likely caused by solid particle aggregation, which makes it more difficult for UV light to penetrate the solution.

A similar behavior to the samples synthesized in this work was observed in the tests conducted on the methylene blue samples with varying concentrations: a decrease in degradation efficiency as the dye concentration increased. According to tests conducted at various pH levels, the commercial TiO_2 sample's degradation percentages were highest at basic pH values. During the half-hour dark period prior to irradiation, the dye solution's adsorption on the catalyst surface was strong at basic pH values.

Overall, comparing the three series of photocatalytic experiments, it can be concluded that the commercial TiO_2 exhibits the highest photocatalytic activity (but comparable with the TiO_2 powder obtained from TiO_2 IP, which contains comparable Rutile – anatase phases), which is in line with the literature, anatase being the most active photocatalytic phase of TiO_2 . Considering the almost neutral

zeta potential of the commercial TiO₂ powder and the negative zeta potential of the two synthesized TiO₂ powders, the influence of pH is important. It can be seen that both synthesized powders are more active at neutral pH while the commercial TiO₂ powder needs slightly alkaline or acidic conditions to activate the photocatalytic activity. Corroborating the electronic microscopy and the photocatalytic data, it is obvious that the size and agglomeration are important but influence to a lesser extent than the phase composition and pH.

4. Conclusions

In conclusion, the commercial sample and the laboratory-synthesized TiO₂ IP sample exhibit comparable photocatalytic activity at neutral pH values. The TiO₂ IP sample shows better photocatalytic activity than the commercial sample at low concentrations of methylene blue (3.2 ppm). At a concentration of 7 ppm, the commercial sample shows a higher efficiency of dye removal, and at a concentration of 10 ppm, the two samples show the same methylene blue degradation percentage. Regarding the TiO₂ NH₂ sample, it was found that it contains mostly rutile, which explains the lower photocatalytic activity compared to the TiO₂ IP and commercial TiO₂ samples. The synthesized TiO₂ IP photocatalyst can be successfully used to degrade emerging pollutants from wastewater, such as dyes, especially at neutral pH, thus avoiding the adverse effects that methylene blue may have on human health and the ecological balance.

In future research, we will investigate the effect of incorporating TiO₂ into the pores of mesoporous silica.

Author Contributions

Conceptualization, A.-M.F., M.N.-L. and A.F.; methodology, A.-M.F., R.-D.T., C.C., L.M. and O.-C.O.; validation, D.F., M.N.-L. and A.F.; formal analysis, R.-D.T., L.M., C.C., O.-C.O.; investigation, A.-M.F., and D.F.; writing—original draft preparation, A.-M.F., writing—review and editing, D.F., M.N.-L. and A.F.; supervision, A.F.; funding acquisition, D.F., M.N.-L. and A.F. All authors have read and agreed to the published version of the manuscript.

Data Availability Statement

Data supporting the findings of this study are available upon reasonable request from the corresponding author.

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Conflicts of Interest

The authors declare no conflict of interest.

Artificial Intelligence Disclosure

No artificial intelligence-based or generative tools were used in the preparation of this manuscript.

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