Titanium dioxide, as environmental (decontamination) photocatalyst, is increasingly gaining importance in the removal of organic pollutants from water and air, as well. Different methods can be used for obtaining TiO$_2$ catalyst, but sol-gel and hydrothermal methods are most commonly used for the synthesis and modification of TiO$_2$. The properties of nanostructured materials such as crystal phase, surface features and electronic structures can be changed with doping. In this paper, the influence of Fe and Zn as dopants on the physico-chemical properties of TiO$_2$ catalyst will be presented based on the reported literature data. Usually applied instrumental techniques for the materials characterizations are X-ray diffraction (XRD), SEM, TEM, FTIR and UV-vis spectroscopy. The removal efficiency of various organic pollutants (organic dyes, antibiotics, pesticides, xenobiotics) is determined by the characteristics and dosage of the catalyst, nature and initial concentration of pollutants, pH, oxidants addition, light intensity and wavelength.

Introduction

Titanium dioxide (TiO$_2$) has attracted a lot of attention in recent decades. TiO$_2$, as a wide band-gap semiconductor, shows very good optical, electrical, mechanical and catalytic properties, which makes it an excellent material for environmental refinement. TiO$_2$ can be used for the decomposition of environmental pollutants as a possible alternative to conventional waste water treatment technologies [1]. Titania is highly studied due to its specific characteristics such as high thermal and chemical stability, non-toxicity, chemical and biological inertness, cost-effectiveness, high photocatalytic activity and strong oxidizing/reducing behavior(s). Photocatalytic activity of TiO$_2$ is based on its semiconductor properties [2]. The band gap of titania is about 3.2 eV, and it occurs in three main different polymorphic phases: rutile (tetragonal, 3.02 eV), anatase (tetragonal, 3.2 eV) and brookite (orthorhombic) [3]. Thermo-chemically, the most stable polymorphic phase of these three mentioned phases is rutile. The other two polymorphic phases, anatase and brookite, can be transformed to rutile at elevated temperatures. When the TiO$_2$ photocatalyst is illuminated with light of the corresponding wavelength, where light photon energy should be equal or greater than the energy of the band gap of the semiconductor, photocatalytic reactions are activated. In this way, the electrons (e$^-$) are induced and they migrate from the valence band to the conduction band. In the valence band, positive holes are generated (h$^+$), and electrons (e$^-$) migrate to the conduction band. Therefore, in the semiconductor, under the influence of light, electron/hole pairs are formed [4,5].

Due to the wide band gap of titania, its use is highly limited because of the necessity for the UV-radiation application in photocatalytic processes in environmental treatment processes [6]. Scientists have tried in various ways to adjust the band gap, and thus enable the activation of TiO$_2$ by using the visible part of the spectrum. These different ways can be: the addition of electron donors (so called: hole scavengers), the addition of transition metal ions (in)to titania, sensitizing using dye molecules, etc. [6].

Low photocurrent efficiency of TiO$_2$ could be overcome by the synthesis of ordered structures of TiO$_2$ or by doping of TiO$_2$ nanoparticles with transition metal ions [4,7]. Iron is a very interesting transition metal which was often used as dopant, and a large number of papers have been published on this subject. Also, Qiao et al. [8] have proven that a small amount of Zn$^{2+}$ ions improved the separation of the charges, while the possibility of recombination significantly reduced [8].

This paper is a short review on published researches on the synthesis of TiO$_2$-based nanopowders, physico-chemical properties and the application in selected photocatalytic reactions.

Titania-based material preparation

Selection of precursors

Most commonly used substances as precursors for TiO$_2$ synthesis are titanium tetrachloride (TiCl$_4$), titanium sulfate and then titanium isopropoxide and/or titanium tetrabutoxide.

Keywords: Iron, Hydro-thermal synthesis, Photocatalysis, Sol-gel, Titania, Zinc

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as alcoxide precursors. However, usages of most of these precursors have certain limitations that are proven in practice. For example, TiCl₄ is problematic for handling because of its property to fume easily. The use of alcoxides in synthesis of titania requires strict control of experimental conditions such as air humidity, pH and temperature. It is known that when alkoxide is used as a precursor, an intense hydrolysis of alkoxide is possible in the air so this is a disadvantage of these precursors usage. Additionally, the commercialization of this process is limited due to high prices of alkoxides. When titanium(IV)-sulfate is used as a precursor, TiO₂ obtained in this case shows a lower catalytic activity compared to the TiO₂ catalyst obtained with the use of titanium tetra-isopropoxide and/or titanium chloride as precursors [9,10].

Goswami and Nath Ganguli [9] reported that the use of precursor potassium hexafluorotitanate in obtaining titania showed better properties than other precursors. Potassium hexafluorotitanate is very stable, water soluble, non-corrosive and it’s not toxic for the environment. Moreover, the product itself and its degradation products are nontoxic [9]. Significant improvements in the reported process by using the cited precursor K₃TiF₆ are:

(i) Water is used as the process solvent that greatly reduces the costs of the process, because water is a cheap solvent, it is not toxic and it is ecologically-friendly,

(ii) A “gel”-like matter rather than a simple precipitate is formed,

(iii) After a thermal decomposition of the “gel”-like matter, the precursor material is obtained and in most cases it provides the production of homogeneous oxide material [9].

Different syntheses methods can be used for obtaining of the TiO₂ catalyst such as: sol-gel [2,11-14], hydrothermal [15], solvothermal [14], the ultrasonic procedure, combining a sol-gel method with a hydrothermal treatment, combining a sol-gel method with a solvothermal treatment [14], the micro-emulsion formation procedure [16], the preparation procedure in flame aerosol reactors [17]. Among them, frequently used methods are sol-gel and hydrothermal method, and therefore they will be observed in this mini review paper.

Process parameters in the preparation procedure

The sol-gel method has become an increasingly important process for the synthesis of TiO₂-based catalyst in recent years, and it has many advantages in obtaining some specific materials and biomaterials with outstanding properties as electric, optoelectronic, magnetic and bioactive [4]. This method provides the obtaining of titania-based catalysts in the form of powder(s) and thin films, as well. In addition, it is possible to control the size of particles and surface properties of the catalyst by using a sol-gel synthesis method. This is a relatively simple method for the synthesis of catalysts/materials with a relatively high chemical purity and homogeneity without the additional use of high temperature treatments or pressure [18]. Organic and inorganic precursors can be used in sol-gel synthesis route. In both cases, mono-dispersions powders characterized with submicron-sizes can be obtained. In the case when inorganic precursors such as titanile sulfate (TiOSO₄) or titanium chloride (TiCl₄) are used in the presence of sodium sulfate (Na₂SO₄) [19,20], or tartaric acid [21], after the thermo(hydro)lysis, in both cases, particles of spherical shapes are obtained with diameters around 0.4 nm. However, organic precursors for obtaining TiO₂ are significantly more frequently used. The precursor undergoes two main reactions: hydrolysis and condensation. Hydrolysis and polycondensation occur at the same time as competitive reactions. There are many factors that can change a relative rate of the mentioned reactions, and the most important factors are: a type of the precursor and solvent, the ratio of water to the catalyst precursor, pH value, temperature, and a presence of additives [22].

Ward and Ko [22] showed that the sol-gel method is a very useful method for obtaining catalytic materials. Because of great potential for the use of titania and corresponding products in various areas, Ward and Ko have worked to improve this method, and thus improve the physical and chemical properties of these materials [22]. They obtained TiO₂ gels by dissolving sodium titanate in concentrated HCl, after which they added K₂CO₃, (NH₄)₂CO₃ or Na₂CO₃ to avoid significant changes in pH, or by using thermo(hydro)lysis of TiCl₄ or TiO(NO₃)₂ under acid conditions. Depending on pH value and the nature of the counter-ions, in this way the colloidal particles obtained at same temperatures may have an anatase or a rutile crystal structure [22,23].

Lately, most researches are based on the use of alkoxides Ti(OR)₄ as precursors for the sol-gel synthesis of TiO₂. For the synthesis of TiO₂ gels, Crisan et al. [24] used alkoxides Ti(OR)₄ as precursors, where R is an organic radical such as ethyl (Et), propyl (n-Pr), isopropyl (i-Pr), n-butyl (n-Bu), and secondary butyl (s-Bu). Further, in the sol-gel synthesis substoichiometric hydrolysis ratios and inorganic acids such as HCl and HNO₃ are used. In this way, catalysts with uniform particles and with a large specific surface area are obtained [24].

Goswami and Nath Ganguli [9] have proposed precursor potassium hexafluorotitanate for the production of TiO₂, the usage of which showed much better properties. These researchers dissolved K₃TiF₆ in distilled water and after that, this aqueous solution hydrolyses and formed a precipitate after the treatment with NH₃·OH solution to pH 7.5 under constant stirring, in a Teflon baker, at room temperature. The resulting precipitate was filtered and washed with distilled water to a negative reaction to fluoride ions. After that, the precipitate was dispersed in hot water and the addition of 10% HNO₃ solution caused peptization. Hydrothermal treatment was performed in a Teflon lined stainless steel autoclave at the appropriate temperature. In this way, the TiO₂ catalyst with the (relatively) good specific surface area, anatase crystal phase and 3.19 eV band gap is obtained [9].

Apart from the sol-gel method, a hydrothermal method for the synthesis of TiO₂ is also often used. Hydrothermal synthesis can be defined as a heterogeneous chemical reaction in the presence of an aqueous solvent, at elevated temperatures and pressures, in a closed system. This method can also be used for treatments of compounds that are relatively insoluble or unstable in water/air under ordinary/normal conditions. The most commonly used precursors in
this synthesis are titanium-based alkoxides. They are activated with acids or bases before the thermal treatment in autoclaves [25,26]. In their research, Byrappa and Adschiri [26] concluded that with the use of hydrothermal method TiO$_2$ powder can be obtained that is characterized with different microstructures, morphologies and different crystal phase compositions by varying the following parameters: temperature, pressure, the duration of the process, the concentration of precursors and pH [26].

Lu et al. [27] have studied the effect of pH on the features of TiO$_2$ powder obtained using a hydrothermal method, and they concluded that in the acidic pH range (pH<6) mixed phases of TiO$_2$, brookite and anatase were obtained; on the contrary, if the conditions were alkaline, a pure anatase form of the TiO$_2$ was obtained by using different temperatures below 230 °C [27]. Phan et al. [28] have proved in their research that using a simple hydrothermal method in the presence of concentrated hydrochloric acid, the control of morphological properties of the obtained TiO$_2$ can be carried out; in case when the preparation started from metallic Ti and hydrogen peroxide over hydrothermal oxidation, ultrafine powders of titania can be obtained [28]. In addition to numerous advantages mentioned above, a negative side is the limitation on the exclusive preparation/production of oxide powders.

Modification of TiO$_2$

Doping is one of the most effective way(s) to change the properties of nanostructured materials such as crystal phase, surface and electronic structures. Doping is the process of the controlled addition of impurities, mainly ionic species in very small quantities into the corresponding base material. If a large amount of dopant ions is added to the material, the crystalline structure of the material can be disturbed, and a second crystal structure or mixed structures can be formed [29]. A group of scientists announced that the addition of various transition metal (M) ions to titania is the best way to adjust/reduce the band gap, as well as the increase of the specific surface area of the nanostructured TiO$_2$. Also, the addition of transition metal ions improves the photocatalytic activity of TiO$_2$ that is an important purpose of doping [6]. For the purpose of gaining a highly active TiO$_2$ catalysts, various transition metals are used as dopants such as: Fe, Ag, Cu, Zn, Cr, Mn, and Co ions [18]. In this mini review paper, the results obtained in various studies on the influence of iron and zinc as dopants of TiO$_2$ will be presented.

A group of authors synthesized titanium dioxide nanopowders doped with different amounts of Fe$^{3+}$ ions by a modified acid-catalyzed sol gel method in non-aqueous medium. Sols were mechanically stirred for 10 minutes, sealed and placed at room temperature. After 5 days, gels were dried at 60 °C for 24 h and then the samples were calcined at 400 °C for 2 h [7]. All the iron-doped catalysts samples obtained in this way have lower band gap values compared to pure anatase (characterized with 3.2 eV). According to the results of these scientists, doping with Fe$^{3+}$ ions increases the adsorption in a visible light zone. Also, the degradation rate of pollutants increases with increasing the amount of iron ions used as dopants [7].

Seabra et al. [2] used titanium(IV)-isopropoxide, zinc nitrate hexahydrate and iron nitrate nonahydrate as precursors for the sol-gel synthesis. Undoped and doped TiO$_2$ powders were prepared in the following manner: titanium isopropoxide and the solution of absolute ethanol were sonicated and then distilled water was added. After sonication, ammonium hydroxide was added and sonicated again. Doped TiO$_2$ powders were prepared in the same way. The iron and zinc precursors were dissolved in the chosen amount of distilled water. The final solutions were centrifuged, after that the obtained powders were dried and calcined at 500 °C. In the structure of un-doped TiO$_2$, after the thermal treatment, anatase was the main crystalline phase, but the presence of the rutile phase was also detected. When doped TiO$_2$ samples were analyzed by XRD technique (Fe and Zn-doped TiO$_2$), anatase was the only detected crystalline phase. Also, Fe and Zn doped titania-based powders were more uniform in size and characterized with smaller particles sizes than undoped TiO$_2$ powders. As far as photocatalytic activities were concerned, it was observed that the photocatalytic activity of the catalyst was reduced if Fe was used as a dopant. However, titania doped with zinc showed a significantly higher photocatalytic activity [2].

Delekar et al. [6] have successfully synthesized a pure anatase phase of iron-doped TiO$_2$ nanoparticles by a simple sol-gel method at room temperature. In this manner, synthesized and modified TiO$_2$ showed an improved absorption of a visible light under UV-vis irradiation, the photocatalytic reaction was effectively initiated. Fe-doped TiO$_2$ showed the improved photocatalytic activity in comparison to pure TiO$_2$ due to lower band-gap energy [6].

Physico-chemical characterization(s)

In order to characterize the synthesized titania-based materials, various techniques can be used such as: X-ray diffraction (XRD) technique, transmission electron microscopy (TEM), scanning electron microscopy (SEM), Fourier-transformed Infrared Spectroscopy (FTIR).

Using the X-ray diffraction (XRD) technique, the phase composition of the synthesized TiO$_2$ nanopowders can be determined, after which the assessment of microstructural parameters can be performed: lattice parameters, crystal size, etc. [30]. XRD patterns of the chosen obtained TiO$_2$ powders are shown in Figure 1. As shown in Figure 1., into the un-doped TiO$_2$ powders calcined at 500 °C, anatase is the main crystalline phase, while the presence of rutile can also be detected in smaller volume fractions. However, in titania-based samples doped with iron and zinc, the only detected crystalline phase was anatase, while a rutile crystal phase was not registered. When iron was used as a dopant into/onto titania, the published results showed a decrease of the powder crystallinity (inset in Figure 1.) [2].
Figure 1. XRD spectra of titania-based powders [2]

Figure 2. SEM images of pure TiO\textsubscript{2} and titania doped with Zn and Fe [2]

TEM provides information about the material morphology at higher resolution, while SEM can be used in characterization of the material morphology at smaller resolution. A group of scientists, Seabra et al. [2] have synthesized TiO\textsubscript{2} powders by the sol-gel route and the particle sizes of the prepared powders were submicron (Figure 2.). The undoped titania showed the bi-modal distribution of particles sizes: fine particles (20-40 nm) together with grainier/bigger ones (300-500 nm). The particles of Zn and Fe-doped TiO\textsubscript{2} samples were characterized with smaller particles (<40 nm) and more uniform in size than the particles of undoped samples. Therefore, the specific surface areas (SSA) of the doped titania-based materials were higher than of the undoped TiO\textsubscript{2} materials. On the other side it has also been reported that doped TiO\textsubscript{2} powders tended to agglomerate with was more evident for the iron-doped titania (Figure 2.) with agglomerates around 1000 nm in diameter. The specific surface area after doping increased from 7 m\textsuperscript{2}/g for undoped TiO\textsubscript{2} to 15 m\textsuperscript{2}/g and 41 m\textsuperscript{2}/g for Fe and Zn doped TiO\textsubscript{2}, respectively [2].

In Figure 3, the FT-IR spectra of 0.0 mol %, 1.0 mol % and 3.0 mol % Fe to bare TiO\textsubscript{2} samples are shown. All samples have been characterized with similar FTIR spectra. A broad peak observed between 3500 and 2800 cm\textsuperscript{-1} is assigned to a stretching vibration mode of the −OH group on the surface titanium centers of TiO\textsubscript{2}. The anatase TiO\textsubscript{2} sample has been characterized with a broad band in the 400-900 cm\textsuperscript{-1} region, and in the investigation of this group of scientists, the FTIR spectra of all titania samples match with those spectra typical for the anatase titania crystal phase [6].

Figure 3. FTIR spectra of TiO\textsubscript{2} and Fe-doped TiO\textsubscript{2} nanoparticles [6]

UV-vis spectra of the representative Fe-doped TiO\textsubscript{2} samples are shown in Figure 4. Delekara et al. [6] have successfully synthesized a pure anatase phase of iron-doped TiO\textsubscript{2} nanoparticles with the improved photocatalytic activity in comparison to pure TiO\textsubscript{2} because of lower band-gap energy [6]. Doping of iron into the TiO\textsubscript{2} lattice provided a lower band gap. The increase of absorption in a visible region with a decrease in a band gap can be assigned to the formation of dopant (energy) level near to the valance band. The determination/estimation of optical properties of the doped titania-based samples resulted in the color change of doped powders from light to dark yellow. The presented results showed that photocatalytic performance of doped catalysts increased with the increase of the dopant amount (Figure 5.) [6].
Figure 4. UV-vis spectra of representative Fe-doped TiO₂ samples [6]

Figure 5. Change in the RB concentration after the use of suspension of iron into/onto TiO₂ nanoparticles [6]

Application of TiO₂ in photocatalysis

Photocatalysis gained a lot of attention in the environmental protection in the field of pollutants degradation/conversion due to a growing need to develop new methods of the pollutants treatment in waste waters and/or air. Titania has a very important place among other photocatalysts because of its high photocatalytic activity, high chemical stability, thermal stability, excellent functionality and finally, it is a cost-effective catalyst. In the recent period, TiO₂ has become an environmental photo-catalyst for a large variety of organic pollutants such as: various organic dyes (Bromocresol green, Azo dye, Orange II, Crystal violet), pesticides, antibiotics, xenobiotics (nitroaromatic compounds) identified in wastewater and air due to the industrial activity [2,6,7,9,12,14]. Seabra et al. [2] examined photocatalytic degradation of Orange II solutions in the presence of TiO₂ powders. In their research, they have concluded in their research that maximum decolouration attained in the case of the use of undoped TiO₂ was 81%, in the use of Zn doped titania 98% and when Fe doped titania was used it was 43%. The Fe doped titania exhibited a tendency to agglomeration and this fact explained a decrease of photodegradation efficiency in that reported case [2]. A group of scientists investigated a photocatalytic conversion of p-nitrobenzaldehyde to p-nitrobenzoic acid using undoped and doped TiO₂. The results showed that there was no conversion of p-nitrobenzaldehyde if the solution was irradiated in the absence of the catalyst, but the photocatalytic performance of the doped catalysts increased with the increase of the dopant amount (they used 1 mol. %, 2 mol. % and 3 mol. % Fe to TiO₂). The maximum conversion of p-nitrobenzaldehyde to p-nitrobenzoic acid for 3 mol. % Fe doped titania was attributed to the lowest band gap energy, the increase in the surface area and higher hydroxyl concentrations on the surface of the oxide surface [6].

The main parameters that affected the efficiency and rate of pollutant degradations are: characteristics and concentrations/dosages of the catalyst, the nature and concentration of pollutants, pH, oxidants addition, light intensity and wavelengths [31].

Influence of pH

pH is an important parameter that affects the photocatalytic degradation of organic pollutants. The value of pH determines the charging of the surface of the photocatalyst, potential hydrophobicity, (net)surface charge of pollutants and the amount of generated hydroxyl radical. A group of scientists have been studied the effect of pH on the degradation of nitrobenzene using TiO₂ suspension. They found that pH did not significantly affect benzene degradation, because unmodified benzene did not react with acid or base and was not ionized under different pH values [32]. In this study, the literature data showed that the maximum removal of organic pollutants was reached at pH between 6.5 and 7. In this range of pH, the pollutant, its degradation intermediates and the used catalyst were neutral; this situation favored the reactions of degradation and mineralization [33].

Influence of Light Intensity and Wavelength

A group of scientists organized comparative studies of photocatalytic degradation of nitrobenzene by using concentrated solar radiations or UV light in the same reactor [33]. The results showed that by using artificial UV lamp for 1h, nitrobenzene was completely removed, but it took 4h to remove nitrobenzene by using solar irradiation. UV lamps can be replaced with solar light by using titania doped with transition metal(s) (Fe, Cu, Zn) with the improved photocatalytic activity in a visible region; this suggested that waste water refining can drastically reduce the water cost and treatments costs [34].
Conclusions

Due to unique characteristics like a photocatalytic activity, excellent functionality, thermal stability, non-toxicity and cost-effectiveness, TiO$_2$ is considered as the most thoroughly investigated semiconductor used as a photocatalyst in the literature. The sol-gel and hydrothermal methods are frequently used for the synthesis and also for modification of TiO$_2$. Titania can be doped with metals, nonmetals and/or ionic components in order to enhance its photocatalytic activity and to extend the response into the visible domain of irradiation. In recent years, various transition metals have been used as dopants such as iron and/or zinc, and it has been proved that after doping of TiO$_2$ under certain conditions, its properties can be significantly improved. A large number of researches confirmed that TiO$_2$-assisted photocatalysis is an efficient method for degradation of different organic pollutants which can be applied for the conversion of toxic organic pollutants into less or non-toxic compounds.

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List of abbreviations

TiO$_2$ - Titanium dioxide  
TiCl$_4$ - Titanium tetrachloride  
K$_2$TiF$_6$ - Potassium hexafluorotitanate  
TiO$_2$SO$_4$ - Titanite sulfate  
Na$_2$SO$_4$ - Sodium sulfate  
K$_2$CO$_3$ - Potassium carbonate  
(NH$_4$)$_2$CO$_3$ - Ammonium carbonate  
Na$_2$CO$_3$ - Sodium carbonate  
TiO(NO$_3$)$_2$ - Titanium nitrate  
Ti(OR)$_4$ - Titanium alkoxides  
HCl - Hydrochloric acid  
HNO$_3$ - Nitric acid  
NH$_2$OH - Ammonium hydroxide  
XRD – X-ray diffraction  
SEM – Scanning electron microscopy  
TEM – Transmission electron microscopy  
FTIR – Fourier-transformed Infrared Spectroscopy  
RhG – Rhodamine 6G

References


TITAN(IV)-OKSIDA U FOTOKATALITIČKOJ RAZGRADNJI/KONVERZIJI RAZLIČITIH ORGANSKIH POLUTANATA: KRATAK PREGLED

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Izvod

HEMIJSKE MODIFIKACIJE NANOSTRUKTURNIH MATERIJALA NA BAZI TITAN(IV)-OKSIDA U FOTOKATALITIČKOJ RAZGRADNJI/KONVERZIJI RAZLIČITIH ORGANSKIH POLUTANATA: KRATAK PREGLED

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Titovni (IV) oksid, kao fotokatalizator koji se može primenjivati za dekontaminaciju životne sredine, sve više dobija na značaju u uklanjanju organskih polutanata iz vode i vazduha. Za dobijanje TiO₂ katalizatora se mogu koristiti različite metode, ali su sol-gel i hidrotermalna metoda najčešće korišćene za sintezu i modifikaciju TiO₂. Osobine nanostrukturnih materijala kao što su: kristalne faze, površinska svojstva i elektronska struktura se mogu izmeniti dopiranjem. U ovom radu će biti razmatrana, na bazi literaturnih podataka, uticaj Fe i Zn kao dopanata na fizičko-hemijske osobine TiO₂. Instrumentalne tehnike koje se najčešće koriste za karakterizaciju materijala su: XRD tehnika, SEM, TEM, FTIR i UV-vis spektroskopija. Efikasnost uklanjanja različitih organskih polutanata (organskih boja, antibiotika, pesticida, ksenobiotika) zavisi od karakteristika i doze/količine katalizatora koji se primenjuje, prirode i inicijalne koncentracije polutanta, vrednosti pH, upotrebe oksidanasa, intenziteta elektromagnetnog zračenja i talasne dužine.