

## Enhancement of the activity of TiO<sub>2</sub>-based photocatalysts: a review

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Titania is one of the most widely used standard reference photocatalysts in the field of environmental applications. However, the wide anatase band gap and high degree of recombination between photogenerated charge carriers is limiting titania overall photocatalytic efficiency in water and air purification. This paper reviews recent studies on semiconductor photocatalysis, carried out at the Institute of Catalysis of the Bulgarian Academy of Sciences, aimed at enhancing the efficiency of TiO<sub>2</sub>-based photocatalysts and obtaining novel photocatalytic systems for sunlight utilization and application of methods improving the overall performance of the photocatalytic processes.

**Keywords:** semiconductor photocatalysis, photochemical oxidation, water purification, environmental remediation, solar light.

### INTRODUCTION

In parallel with the ever-growing amount of persistent organic contaminants accumulating in air, wastewaters, and soil, the ecological legislation and the respective normative restrictions are becoming more and more severe [1]. In many cases, standard oxidation processes (biological, chemical, or physical) for removal of pollutants are still economically unfavourable; moreover, they do not achieve complete mineralization of organic and inorganic pollutants [2]. One of the modern methods for removal of organic and inorganic compounds, persistent towards conventional means of neutralization, is destruction by applying the so-called ‘advanced oxidation processes (AOPs)’. These processes are based on generation of highly reactive particles of radical type, mainly HO• radicals, which destroy a large variety of organic and inorganic chemical compounds [3].

Among the AOP processes, heterogeneous photocatalysis appears to be one of the basic and most efficient approaches for degradation of organic contaminants in water, air, and soil [4]. Various oxides, chalcogenides, and composite materials and alloys are finding application in classical heterogeneous photocatalysis manifesting semiconductor properties. One of the most intensively studied photocatalysts is titanium dioxide (TiO<sub>2</sub>) [5], in whose presence photocatalytic processes occur under UV light irradiation, however, a high degree of recombination between the photogenerated charge carriers is lowering its efficiency as photocatalyst.

Aimed at overcoming the disadvantages of the photocatalysts based on TiO<sub>2</sub> and promoting the efficiency of the photocatalytic processes some new semiconducting materials are being developed, excited simultaneously by both the UV and visible components of the solar radiation [6], as well as such materials, in which the charges are separated more efficiently [7]. The increase in photonic efficiency of the processes of destruction of water and air pollutants as well as the utilization of solar light for this purpose is driving forward to realization of large-scale projects solving ecological problems and achieving at the same time improvement of the quality of light.

### I. TITANIA-BASED PHOTOCATALYSTS MODIFIED SUPERFICIALLY WITH NOBLE METAL NANOPARTICLES

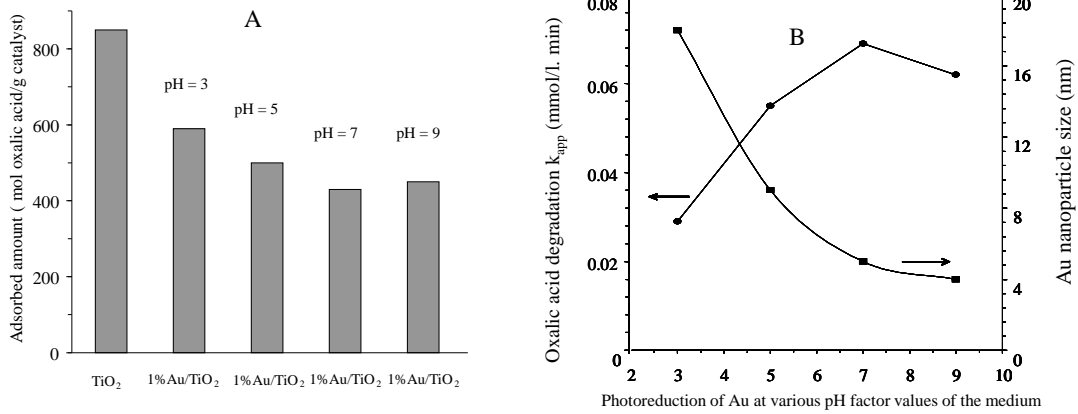
Photocatalysts based on titania were modified superficially (0.1–1 wt.%) with nanoparticles (Ag, Au, Pd, Pt) aimed at increasing the quantum yield of the processes of destruction of organic and inorganic compounds [8–13]. The photocatalysts have been characterized by physical methods.

It was found that the sizes of noble metal nanoparticles attached to the surface of TiO<sub>2</sub> have a substantial influence on their photocatalytic activity. A remarkable influence of the pH of the medium during synthesis on Au particle size was registered upon attaching gold on the titania surface by photo-reduction, which exerts an effect also on adsorption capacity with respect to oxalic acid (Fig. 1A). Thus for one and the same quantity of gold deposited on TiO<sub>2</sub> (1 wt.%), when the size of the nanoparticles was decreased from 18 nm down to 4 nm, the rate

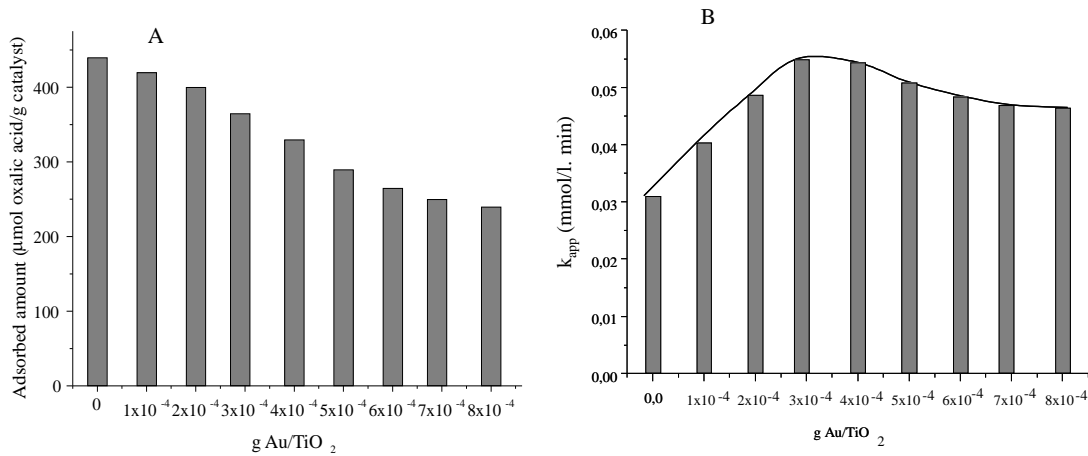
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constants of the photocatalytic destruction of oxalic acid were increased more than twice (Fig. 1B) [11]. It has been shown that the photocatalytic activity is affected by the quantity of metal nanoparticles attached on the surface of TiO<sub>2</sub> (Fig. 2) [10]. The reason for this is that two factors acting in opposite

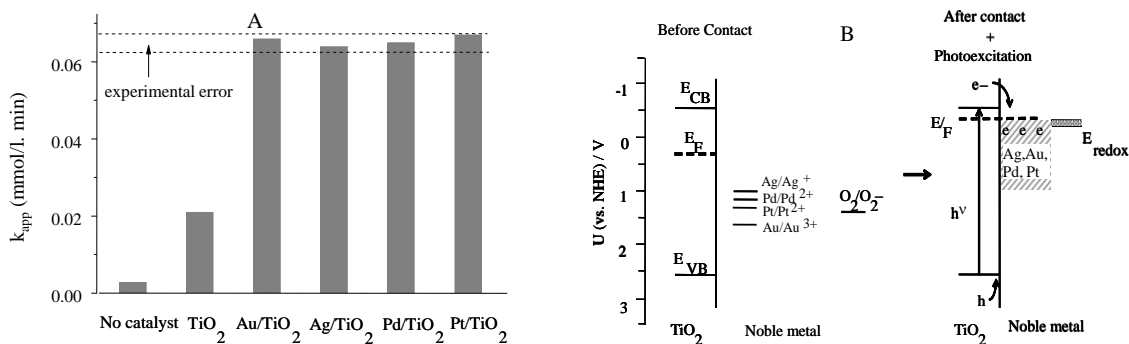
directions are influencing photocatalyst efficiency: (i) the metal nanoparticles improve charge carrier separation and (ii) they decrease the adsorption capacity of TiO<sub>2</sub> because of partial blocking of adsorption sites on its surface.



**Fig. 1.** A – total amount of oxalic acid adsorbed at pH 3 on TiO<sub>2</sub> photocatalysts prepared at various pH factor values of the medium; B – dependence of the rate constants of photocatalytic degradation of oxalic acid on Au<sup>0</sup> nanoparticle size.



**Fig. 2.** A – total amount of oxalic acid adsorbed on TiO<sub>2</sub> at pH 3 and on Au-modified TiO<sub>2</sub> materials; B – dependence of the rate of photocatalytic degradation of oxalic acid on gold loading.



**Fig. 3.** A – dependence of the rate constants of photocatalytic degradation of oxalic acid on the nature of the noble metal particles on TiO<sub>2</sub>; B –Fermi level energies of the TiO<sub>2</sub>/noble metal composites upon photoexcitation.

It was also ascertained that the chemical nature of the noble metal attached on the surface of TiO<sub>2</sub> does not influence photocatalyst efficiency [13]. In this aspect at the same size of noble metal nanoparticles ( $\approx 5$  nm), deposited on the surface of TiO<sub>2</sub>, the rate constants of destruction of the pollutants in water are approximately the same (Fig. 3A).

Photo-physical investigations on titania/noble metal systems explain the obtained results. It has been shown [14] that as a result of equilibrated process of electron transfer from oxide semiconductors to the metal nanoparticles the life-time of the photoexcited electron is increased in the conduction band of the semiconductor (Fig. 3B). The lifetimes of the excited states are approximately the same in cases of attaching Ag, Au, Pd or Pt on the semiconductor [15], and for this reason the photocatalytic activity is varying within the framework of the experimental error (Fig. 3A) [13].

## II. INORGANIC OXIDES WITH SEMI-CONDUCTING PROPERTIES: PHOTOCATALYSTS FOR UTILIZATION OF SOLAR LIGHT

Two types of photocatalytic systems have been prepared and studied, which are able of being excited simultaneously under irradiation with UV and visible light. One type of systems is coupled composites of two oxides: one with a wide band gap and another one with a narrow band gap [16–18]. The second type of materials is prepared by doping the titania and a WO<sub>3</sub>/TiO<sub>2</sub> composite with nitrogen by means of anionic replacement of oxygen atoms by nitrogen entities in the crystal lattices of TiO<sub>2</sub> or WO<sub>3</sub>) [19–21]. The photocatalysts have been characterized by physical methods.

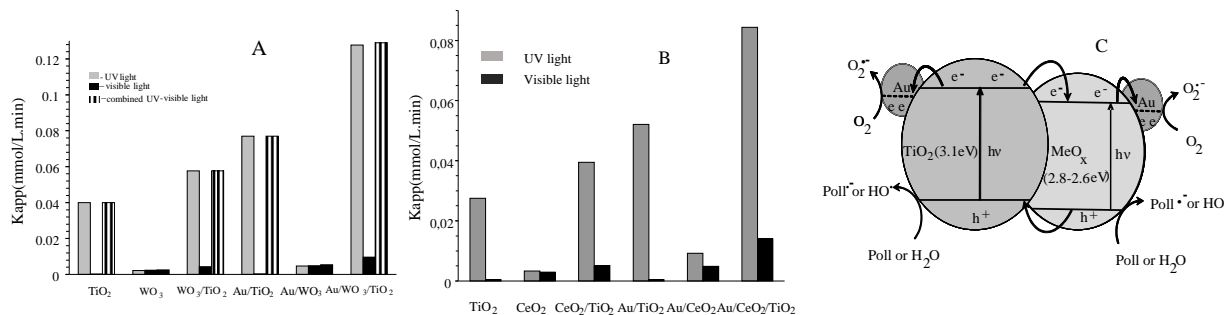
### II. 1. Coupled semiconductors

The photocatalytic activity of WO<sub>3</sub>/TiO<sub>2</sub> and CeO<sub>2</sub>/TiO<sub>2</sub> composites was studied in the degrada-

tion of 4-chlorophenol and oxalic acid under irradiation with visible [16] and ultraviolet light in separate and under combined UV-vis light irradiation [17,18]. The photocatalytic oxidation of both contaminants occurs completely leading to total mineralization: formation of CO<sub>2</sub>, H<sub>2</sub>O, and HCl. The band gap of anatase TiO<sub>2</sub> is about 3.2 eV and because of that, it is excited only by UV light. However, the band gaps of WO<sub>3</sub>  $\approx 2.8$  eV and CeO<sub>2</sub>  $\approx 2.75$  eV enable their photo-excitation by visible light, i.e. photons of energy  $E_{hv} \geq$  higher or equal to the energy of the band gap of the semiconductor.

It has been observed that under irradiation with UV light of the coupled photocatalyst systems the rate constants of destruction of oxalic acid are much higher than those registered under irradiation with visible light (Fig. 4A). Visible light illumination of WO<sub>3</sub>, CeO<sub>2</sub> and of the binary composites WO<sub>3</sub>/TiO<sub>2</sub> and CeO<sub>2</sub>/TiO<sub>2</sub> does not lead to high activity due to a small amount of tungsten or cerium oxides in the composites (4–6 wt.%) [17,18].

Under irradiation with UV or visible light the rate constants of destruction of oxalic acid or 4-chlorophenol photocatalysed by WO<sub>3</sub>/TiO<sub>2</sub> or CeO<sub>2</sub>/TiO<sub>2</sub> are higher than those registered with the individual oxides (Figs. 4A,B). This fact can be attributed to more efficient separation of surface charge carriers, i.e. to an increase in the quantum yield of the formed HO<sup>•</sup> radicals in the valence bands of both components in the composite photocatalyst (Fig. 4C). Thus under irradiation with UV light the rate constant of the process of oxalic acid destruction catalysed by WO<sub>3</sub>/TiO<sub>2</sub> is  $\sim 1.5$  times higher than that on pure titania [17], while in the case of photo-oxidation of 4-chlorophenol under irradiation with visible light it is 2.5 times higher than that on pure tungsta [16]. Under UV-A light irradiation the CeO<sub>2</sub>/TiO<sub>2</sub> photocatalyst showed a 0.44-fold increase in the rate of oxalic acid mineralization compared to that over the TiO<sub>2</sub> photocatalyst [18].



**Fig. 4.** Dependence of the apparent rate constants  $k_{app}$  of photocatalytic degradation of oxalic acid on irradiation type: A – WO<sub>3</sub>/TiO<sub>2</sub> photocatalyst; B – CeO<sub>2</sub>/TiO<sub>2</sub> photocatalyst; C – schematic representation of charge carrier separation in the photoexcited photocatalysts.

Modifying the surface of the studied semiconductors with Au nanoparticles is leading to a manifold increase in the rate constants of destruction of 4-chlorophenol or oxalic acid both in the cases of UV light irradiation and visible light irradiation (Figs. 4A,B). The enhancement of the photocatalytic activity is owing to a more efficient separation of the charge carriers in the photoactivated catalysts thus leading to an increase in the quantum yield of formation of the HO<sup>•</sup> radicals (Fig. 4C). Especially efficient is the charge separation in the case of Au/WO<sub>3</sub>/TiO<sub>2</sub> photocatalyst under irradiation with UV or combined UV-visible light, where the rate constants of destruction of the oxalic acid are approximately 1.7 times higher than that of the process catalysed by Au/TiO<sub>2</sub> and 3 times higher than that catalysed by pure titania [17]. In the case of photocatalytic destruction of 4-chlorophenol, occurring under irradiation with visible light, the rate constant of the process catalysed by Au/WO<sub>3</sub>/TiO<sub>2</sub> is 3 times higher than that of the process catalysed by tungsta [16]. Particularly efficient is the charge separation process in the Au/CeO<sub>2</sub>/TiO<sub>2</sub> material upon irradiation with UV light (Fig. 4B). In this case, the rate constants of oxalic acid decomposition were approximately twice higher than those over the Au/TiO<sub>2</sub> material and three times higher than those catalysed by pure titania.

## II. 2. Nitrogen-doped semiconductors

Nitrogen-doped titania (N-TiO<sub>2</sub>) and tungsta/titania (N-WO<sub>3</sub>/TiO<sub>2</sub>) catalysts have been synthesized using sol-gel technology. XPS studies established that the atomic concentration of the nitrogen atoms in the crystal lattices of TiO<sub>2</sub> and WO<sub>3</sub>/TiO<sub>2</sub> is  $\approx 1.2\%$ . All the photocatalysts have been additionally modified superficially with gold nanoparticles (0.5–1 wt.%) by the method of photo-reduction.

The photocatalytic destruction reactions of 2-propanol, oxalic acid, and 2,4,6-trinitrotoluene (TNT) in the presence of TiO<sub>2</sub>, N-TiO<sub>2</sub>, WO<sub>3</sub>/TiO<sub>2</sub>, N-WO<sub>3</sub>/TiO<sub>2</sub>, and superficially modified with Au nanoparticles photocatalysts have been investigated under irradiation with UV, visible, and combined UV-visible light [19–21]. It was proved that there occurs a complete destruction of the substrates, which is due most of all to the generated HO<sup>•</sup> radicals.

The ability of nitrogen-doped titania to be photo-excited simultaneously under irradiation with UV and visible light is owing to the fact that, in case of replacing the oxygen atoms in the crystal lattice of TiO<sub>2</sub> by nitrogen atoms, new admixture electron

energy levels are being formed resulting in a narrower band gap. This explains the possibility to excite simultaneously a photocatalyst under UV and visible light irradiation (Fig. 5B).

Concerning the photocatalytic activity of nitrogen-doped catalysts (Fig. 5A) the following conclusions can be drawn.

1. N-doped TiO<sub>2</sub> and N-doped WO<sub>3</sub>/TiO<sub>2</sub> as well as samples modified by nanosized Au particles are efficient photocatalysts for removal of organic contaminants from water under irradiation with UV and visible light.

2. Under irradiation with UV light, the photonic efficiencies of N-doped TiO<sub>2</sub> and N-doped WO<sub>3</sub>/TiO<sub>2</sub> are one order of magnitude higher than the efficiency under irradiation with visible light. This is because of the nitrogen atoms (1.2 at.%) incorporated in the crystal lattice of N-doped photocatalysts. Higher rate constant values for organic contaminants decomposition upon irradiation with UV light are due to the photocatalytic processes occurring simultaneously on N-doped TiO<sub>2</sub>, N-doped WO<sub>3</sub>, and coupled N-doped WO<sub>3</sub>/TiO<sub>2</sub> photocatalysts.

3. An increased photocatalytic activity of N-doped TiO<sub>2</sub> and N-doped WO<sub>3</sub>/TiO<sub>2</sub> under irradiation with visible light is owing to the formation of a new impurity electron energy level leading to narrowing of the band gap of the photocatalysts. Thus, under irradiation with visible light the rate constant values for TNT photooxidation, catalyzed by N-doped WO<sub>3</sub>/TiO<sub>2</sub> or N-doped TiO<sub>2</sub>, are 2-fold and 5-fold higher, respectively, than those for undoped WO<sub>3</sub>/TiO<sub>2</sub> or TiO<sub>2</sub> samples, accordingly.

4. The photocatalytic activities of gold-modified N-doped TiO<sub>2</sub> and N-doped TiO<sub>2</sub>/WO<sub>3</sub> photocatalysts under irradiation with UV or visible light is increased more than twice compared with that of unmodified samples. The higher rate of photocatalytic destruction of the pollutant in the case of deposited nanosized Au particles on the surface of the catalysts is a result of more efficient charge separation, increased lifetime of the charge carriers, and enhanced efficiency of the interphase charge transfer to adsorbed pollutants.

## III. PROMOTING THE EFFICIENCY OF PHOTOCATALYTIC PROCESSES OCCURRING WITH THE PARTICIPATION OF OZONE

Efficiency enhancement of the photocatalytic processes of destruction in aqueous medium has been studied with oxalic acid as pollutant [22] and also with 2,4,6-trinitrotoluene [23] under irradiation with UV-A or UV-C and visible light in the

presence of oxygen-ozone mixture. The concentration of ozone in the mixture was  $6.7 \times 10^{-4} \text{ mol.L}^{-1}$ . A parallel is made in these studies between the efficiency of the photocatalytic processes occurring in the presence of oxygen with that of the processes accomplished involving the participation of ozone. In both cases, it was found that the photocatalytic destruction of the contaminants is reaching a complete decomposition of their organic structure with the formation of CO<sub>2</sub>, H<sub>2</sub>O, and NO<sub>3</sub><sup>-</sup>.

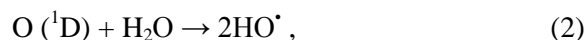
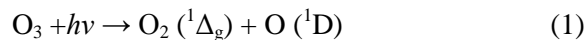
It was shown that in the absence of illumination the ozone does not react directly with the oxalic acid [22], while in the case of TNT some degree of destruction is registered occurring at a very low rate constant (Fig. 6A) as a result of formation of HO• radicals from the ozone in aqueous medium at pH 7 [23].

Under irradiation with ultraviolet or visible light in the presence of ozone the rate constants of destruction of the pollutants are much higher than those registered in the presence of oxygen only and they depend on the illumination intensity of the UV light source [22,23] (Fig. 6A).

A higher efficiency of the photocatalytic pro-

cesses of organic molecules destruction in the presence of ozone is due to generation of HO• radicals from ozone (Fig. 6B) by the following mechanism (equations 1–5):

- Direct photolysis of ozone under irradiation with UV-C light ( $\lambda = 254 \text{ nm}$ )



where O<sub>2</sub> (<sup>1</sup>Δ<sub>g</sub>) is singlet oxygen and O (<sup>1</sup>D) is an oxygen atom in excited state.

- Generation of HO• radicals from adsorbed ozone molecules on the conduction band of the semiconductors



The hydroxyl radicals are among the most reactive free radicals and they are strong oxidizing agents capable of causing destruction of organic compounds.

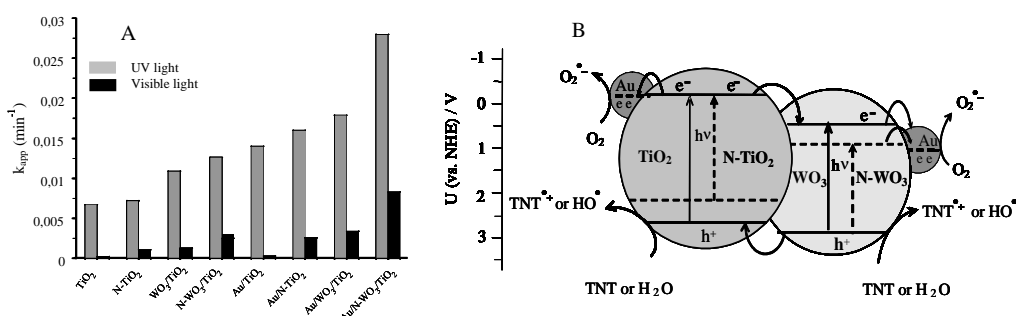


Fig. 5. A – dependence of apparent rate constants of TNT degradation; B – schematic representation of charge carrier separation in photoexcited Au/N-WO<sub>3</sub>/TiO<sub>2</sub> photocatalyst.

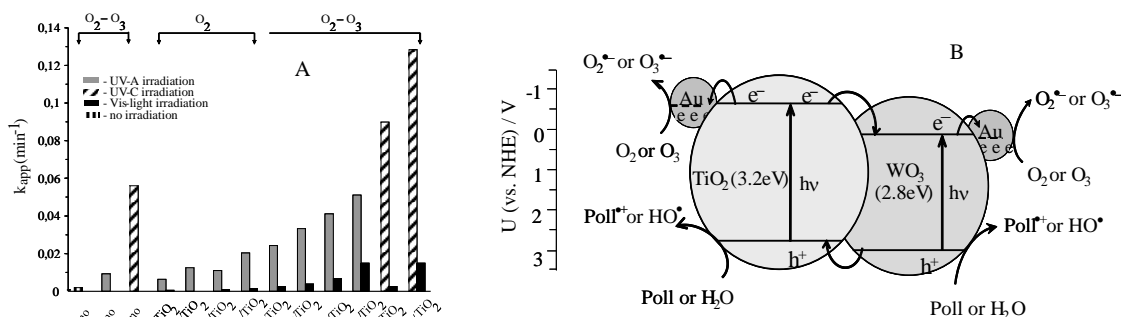


Fig. 6. A – dependence of the rate constants of photocatalytic degradation of TNT under the experimental conditions; B – schematic representation of charge carrier separation in the photoexcited Au/WO<sub>3</sub>/TiO<sub>2</sub> photocatalyst.

It is seen in Fig. 6A that under irradiation with UV light in the presence of ozone the rate constants of TNT photooxidation catalyzed by Au/WO<sub>3</sub>/TiO<sub>2</sub> are 6 times higher, while under visible light irradiation they are 11 times higher than those of the processes occurring in the presence of oxygen only [23]. An analogous tendency of increase in the rate constants (4–14 times) is also observed in the case of photocatalytic destruction of oxalic acid [22]. An additional increase was registered for the rate constants of destruction of TNT in the presence of ozone, which can be interpreted as appearance of synergistic effect [23].

The efficiency of the processes of destruction of TNT in the presence of ozone depends also on the type of photocatalyst (Fig. 6A). Thus in the case of irradiation with UV-A, UV-C, and visible light in the presence of ozone the rate constants of TNT photooxidation catalyzed by Au/WO<sub>3</sub>/TiO<sub>2</sub> are respectively 2.1, 1.4, and 6.2 times higher than those registered with pure titania [23]. Promoting the activity of the studied photocatalysts, Au/TiO<sub>2</sub>, WO<sub>3</sub>/TiO<sub>2</sub>, and Au/WO<sub>3</sub>/TiO<sub>2</sub>, is due to more efficient charge carrier separation during photoexcitation leading to an increase in the quantum yield of HO• radicals' formation (Fig. 6B).

During irradiation with UV or visible light the efficiency of the photocatalytic processes, occurring in the presence of catalysts superficially modified with noble metal nanoparticles, is 1.5–3 times higher than that in the case of non-modified catalysts (Fig. 6A). The increase in the rate constants of destruction of pollutants in water is owing to more efficient charge carrier separation in these materials during photoexcitation (Fig. 6B).

## CONCLUSIONS

Increasing the efficiency of titania-based photocatalysts and enhancing the photocatalytic processes, taken as a whole, is a top priority task for establishing the photocatalysis as one of the basic and most efficient processes of neutralization of the harmful effect of organic contaminants in wastewater, air, and soil. It has been shown that by modifying superficially TiO<sub>2</sub> with noble metals the activity of the catalysts is growing up by 2 to 4 times. Using coupled or doped photocatalytic materials, in addition to promoting the photocatalytic activity, is creating also conditions for more efficient utilization of solar radiation. A considerable growth in efficiency of the photocatalytic processes is registered in the case of accomplishing processes involving ozone as oxidizing agent, whereupon the rate constants of

destruction of the organic contaminants in water is being enhanced by 4 to 14 times.

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## ПОВИШАВАНЕ НА АКТИВНОСТТА НА ФОТОКАТАЛИЗАТОРИ НА ОСНОВАТА НА TiO<sub>2</sub>: ОБЗОРНА СТАТИЯ

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(Резюме)

Титановият диоксид е един от стандартните фотокатализатори използван широко в областта на опазване на околната среда. Въпреки това, широката забранена зона на титановия диоксид и високата степен на рекомбинация на фотогенерираните носители на заряди ограничават неговата ефективност като фотокатализатор при пречистване на води и въздух. В тази статия се прави преглед на последните изследвания върху фотокатализа с полупроводникови материали провеждани в Института по катализ, БАН, целящи повишаване ефективността на фотокатализатори на базата на TiO<sub>2</sub>, получаване на фотокаталитични системи за утилизация на слънчева светлина, както и прилагане на методи влияещи върху повишаване на ефективността на фотокаталитичните процеси като цяло.