ISSN 2079-1704. Хімія, фізика та технологія поверхні. 2025. Т. 16. № 1. С. 141-148

UDC 544.77 + 54.116:631.34 doi: 10.15407/hftp16.01.141

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STRUCTURE, PHOTOLUMINESCENCE, AND PHOTOCATALYTIC ACTIVITY OF $TiO_2@Au/Sm^{3+}$ NANOCOMPOSITES FOR DYES REMEDIATION

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The textile industries produce a lot of toxic dyes and pigments that can absorb and reflect sunlight in the water. The application of heterogeneous photocatalysis for the removal of these pollutants is effective using metal-oxide-based nanocomposites with noble and rare earth metals under different light irradiation. The effect of dopant concentration on the structural, optical, and electronic properties of TiO_2 nanomaterials is also important for their photocatalytic performance. The synthesis of TiO_2&Au/Sm particles with a gold content of 2 and 4 wt. % and samarium 0.2 wt. % was carried out by the chemical precipitation of a TTIP (titanium tetraisopropoxide) solution in the presence of Sm^{3+} and Au^{3+} cations. The primary particle size CSR (coherent scattering region) of nanopowders was 18–20 nm. It has been shown that gold is included in the composite structure, with an increase in the parameters of the anatase crystal lattice. The presence of samarium in the composite structure was confirmed by EDS and PL methods. The obtained structures have shown high sorption activity to cationic dyes (Methylene Blue and Rhodamine B), lower – to anionic ones, in particular, Orange G. It is shown that under the influence of UV irradiation, the solutions undergo decolorization, which is accompanied by the destruction of dye molecules, which is confirmed by the hypsochromic shift of the characteristic maximum in the UV-VL spectra. The series OG (46.7 %) < MO (89.2 %) < MB (96.0 %) < RhB (98.5 %) for TiO_2&Au/Sm particles ([Au] = 2 wt. %, [Sm] = 0.2 wt. %) and MO (22.6 %) < RhB (24.1 %) < OG (41.5 %) < MB (98.6 %) for TiO_2&Au/Sm ([Au] = 4 wt. %, [Sm] = 0.2 wt. %) particles were found according to the intensity of decolorization of the dye solutions. The difference in the course of the photocatalytic process can be related to the PZC (point of zero charge) of the composite particles, which is 7.01 ([Au] = 2 wt. %) and 9.25 ([Au] = 4 wt. %).

***Keywords**: titanium tetraisopropoxide, anatase, samarium, gold, organic dyes, UV irradiation, photocatalysis*

INTRODUCTION

It is well known that titanium dioxide nanoparticles exhibit high photocatalytic efficiency and are successfully used in the photocatalytic destruction of organic dye solutions under UV light [1]. The reasons for limiting the photoactivity of catalysts based on TiO_2 have been widely discussed in numerous scientific publications. The most important disadvantages are related to the band gap's width of polymorphic titanium oxide semiconductors and the high rate of recombination of photoexcited electron-hole pairs on the surface of TiO_2 particles [2]. An effective means of improving the photoactivity of titanium dioxide as well as expanding its photoactivity to the visible light region [3] is doping of TiO_2 structure with elements of a different nature (Ag, Pd, and Au), or the creation of nanocomposites with Fe_3O_4 nanoparticles [4–7].

As a rule, the modification of the metal-oxide surface with metal ions and nanoparticles leads to an increase in the optical and photocatalytic activity of TiO_2. Moreover, the modification of titanium dioxide particles with gold NPs not only reduces the band gap width but also increases optical absorption by systematically shifting the absorption edge into the visible range [8], and gives plasmon resonance with charge separation [9]. Another group of elements that can influence the structure and properties of titanium dioxide particles are rare earth elements [10], among which cerium is the most common [11]. At the same time, several publications have appeared in recent years that demonstrated the prospects of using samarium-loaded titanium dioxide structures for the creation of gas sensors [12], dye-sensitized solar cells [13], as well as the creation of heterogeneous photocatalysts that have been tested for removal of the Rhodamine B dye under the natural sunlight [14] or synthesis of the dye in the Pechmann reaction [15].

In our previous works, we showed the effectiveness of Malachite Green dye destruction in the presence of TiO_2&Au&Gl (glucose) nanoparticles under visible light action [16]. We also presented a comparative study of the destruction of several organic dyes in the presence of TiO_2&Au nanoparticles under UV irradiation [17–18].

This study evaluates the effect of samarium cations on the structure, photoluminescence, and photocatalytic performance of obtained TiO_2&Au nanocomposites by the co-deposition method.

OBJECTS AND METHODS OF THE RESEARCH

Synthesis of TiO_2 nanocomposites doped with Au and Sm cations. The synthesis of nanocomposite powders was carried out by co-precipitation of titanium tetraisopropoxide (TTIP) water-ethanol solutions with standard titers of $HAuCl_4$ and $Sm(NO_3)_3$ solution in the presence of CH_4N_2O (urea) as a hydrolysis regulator, KOH as a nucleating agent, and NH_4OH as a pH value regulator. The amount of gold in titanium oxide powders was set at the level of 2 and 4 wt. % and the part of samarium does not exceed 0.2 wt. %. The resulting precipitates were rinsed with water and ethanol solutions, subsequent lyophilization at $T = 105$ °C and calcinations of the precipitates were performed at $T = 600$ °C within 2 hours.

Physicochemical characterization of TiO_2&Au/Sm nanocomposites. Powders X-ray diffraction study (XRD), Thermal-gravimetric analysis (TG-DTA), Photoluminescence (PL), and Scanning electron microscopy method combined with Energy dispersive spectroscopy module (SEM-EDS) were used to characterize the TiO_2 composite powders.

A DRON 3M diffractometer equipped with a Cu anode tube was used to carry out of a powder XRD analysis. Interpretation of XRD data was performed by the help of the MAUD program. Thermal-gravimetric and differential thermal analysis (TG-DTA) of the precipitates was carried out in a static air atmosphere on a Q-1500D thermal analyzer (Hungary) in the temperature range of 20–1000 °C. A Mira 3 Tescan equipped with an EDX module (Oxford INCA x-act) was used to visualize the nanocomposites, study the morphology, and elemental analysis. He-Cd laser was used at 325 nm for the PL study of powdered samples. The point of zero charge (PZC) of the particles was measured by potentiometric titration.

Photocatalytic activity test of the TiO_2&Au/Sm nanopowders. The photocatalytic activity of the TiO_2&Au/Sm particles was studied through the photodegradation of Methylene Blue (MB), Rhodamine B (RhB), Methyl Orange (MO), and Orange G (OG) with the concentrations of 10 and 20 mg/dm^3. To this end, a nanocomposite powder (100 mg) was added to a dye solution (80 ml) and stirred in the dark for 40 min until the system reached sorption-desorption equilibrium. To study the photocatalytic performance under UV irradiation, a portable Xe 15A lamp (LOT Design, 15.5 mW/cm^2) with a power of 300 W was used. Sampling was carried out after 20, 40, 60 and 80 min of UV irradiation in the suspension mixing mode. The optical density of the solutions was measured on a UV-Vis-NIR Spectrometer (Cary 5000, Agilent) in the range of wavelength from 200 to 800 nm. The concentration of the dyes was found by calibration curves.

The degree of efficient dye's destruction (*D*, %) was estimated according to the standard equation:

$$D, \% = (1\text{-}C/C_0)\cdot 100,$$

where C is the residual concentration of the dye, and C_0 is the initial concentration of the dye.

RESULTS AND DISCUSSION

Thermal-gravimetric analysis. Thermal-gravimetric analysis of precipitate obtained in TTIP system in the presence of 4 wt. % gold and 0.2 wt. % samarium shows the weight loss effect on the DTG curve at 115 °C corresponding to removal of adsorbed-binding water (Fig. 1). The total mass loss is 17.7 %. The endoeffect in DTA curve at 130 °C corresponds to the hydrolysis of TTIP. An intensive exoeffect at 310 °C describes the destruction of TTIP and the combustion of organic components. The formation of anatase structure carries out up to 440 °C. The polymorphic transformations of TiO_2 are fixed at 485 °C (an intermediate β-TiO_2 phase) and at 640–830 °C (rutile phase). Taking into account the low concentration of Au and Sm part the corresponding effects of $Au(OH)_3$ dehydroxylation and the formation of Au_2O_3 (230 °C) are overlapped as well as two endothermic peaks arise from the decomposition of $Sm(OH)_3$ precursors at 457.8 °C and formation of Sm_2O_3 at 671 °C [19].

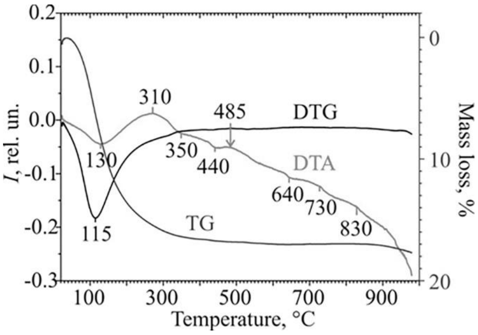


Fig. 1. TG-DTA curves of the titanium(IV) isopropoxide precipitate doped with gold (4 wt. %) and samarium (0.2 wt. %)

X-ray Diffraction Analysis. The phase composition of the TiO_2 powder formed in the presence of gold and samarium admixture and calcined at 600 °C was studied by XRD method. Only anatase phase (PDF No. 21-1272) is determined in Fig. 2. The weight quantity of Au and Sm is relatively low and they do not form separate phases. The crystal lattice parameters, CSR and PZC values of TiO_2 particles doped with gold (2 and 4 wt. %) and samarium (0.2 wt. %) are summarized in Table 1. The increase in the gold part in the structure of the samples leads to an increase in the lattice parameters and a decrease in the CSR. The d (101) distance insignificant shifted to the smaller Bragg angles when the Au part increased from 2 to 4 wt. %.

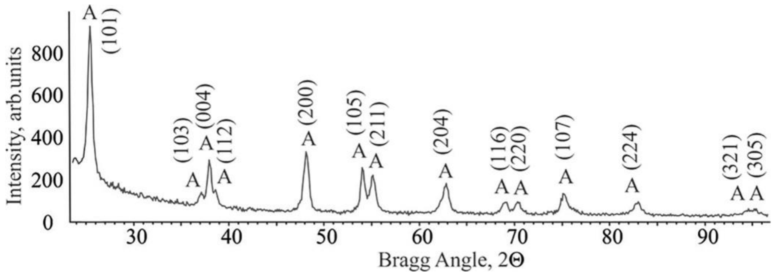


Fig. 2. XRD spectrum of the anatase particles doped with Au (4 wt. %) and Sm (0.2 wt. %)

Table 1. Structural characteristics of anatase nanoparticles doped with gold and samarium cations (600 °C)

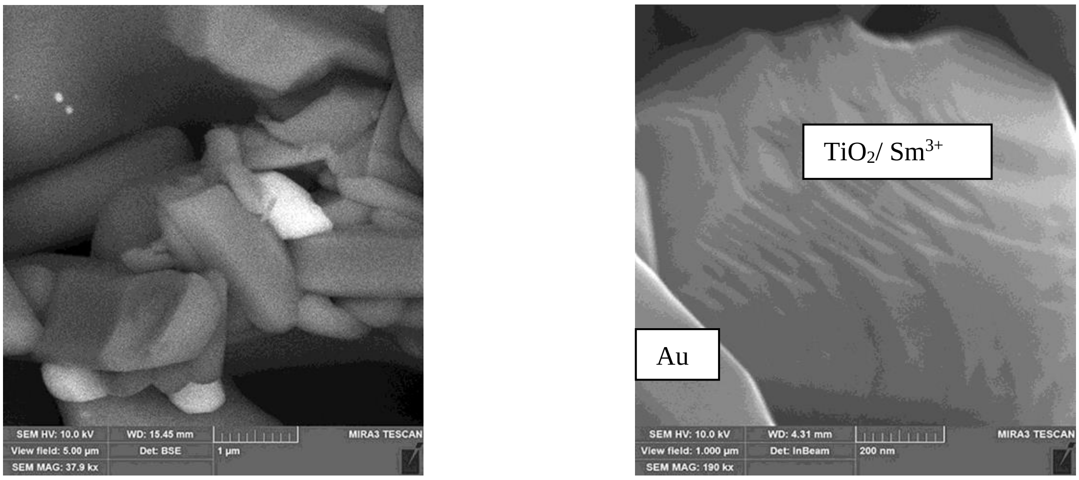
Lattice parameters, nm	***V*, nm^3**	***c/a***	***d* (101), nm**	**CSR, nm**	**PZC**
TiO_2&Au(2.0 wt. %)/Sm					
a = 0.3772	0.135	2.51	0.35040	20.0	7.01
TiO_2&Au(4.0 wt. %)/Sm					
a = 0.3778	0.136	2.52	0.35110	18.2	9.25

Scanning Electron Microscopy. The morphology of the nanocomposites was studied by the scanning electron microscopy. SEM images of TiO_2&Au/Sm ([Au] = 4 wt. %, [Sm] = 0.2 wt. %) sample are presented in Fig. 3, where Fig. 3 *a* – the general view of the powder, and Fig. 3 *b* – the structure of TiO_2 matrix doped by Au and Sm.

Energy Dispersive Spectroscopy. The chemical composition of the TiO_2&Au/Sm powder (wt. %) according to EDS is indicated in Table 2. The main elements are Ti, Au, Sm, and O. Residual K admixture is included in the structure of the powder. Moreover, the Ti : O ratio is less than 2 indicating the location position of modifier ions**.**

Photoluminescence study. The intensity of the PL emission for the TiO_2&Au/Sm sample is suppressed compared to pure TiO_2 (anatase) with a disappearing peak at 450÷570 nm (Fig. 4). This result is evidence of Au nanoparticles capturing photoexcited electrons from TiO_2 semiconductor with the presence of smaller Sm^{3+}dopant and electrons transferred to Au with possible photogenerated holes from TiO_2 [20]. The obtained results demonstrate that the oxygen vacancy in the structure of TiO_2 with Sm^{3+} influences the optical and photocatalytical activity of the TiO_2&Au/Sm nanocomposite. This

PL result is probably due to LSPR (localized surface plasmon resonance) between Au NPs and system TiO_2/Sm^{3+}. In TiO_2&Au/Sm^{3+}, the luminofor is TiO_2/Sm^{3+} and metal is Au NPs. So, under the process of PL emission, Au NPs absorb the energy (emitting light) transferred from TiO_2/Sm^{3+} and quenching of luminescence.



a *b*

Fig. 3. SEM images of TiO_2&Au/Sm: *a* – morphology of the powder (magnification 37900), *b* – surface of anatase matrix doped by Au and Sm^{3+} (magnification 190000)

Table 2. Chemical composition of TiO_2&Au/Sm according to EDS Data

Spectra	**Elements, wt. %**				
	O	**Au**	**K**	**Sm**	**Ti**
1	56.47	3.91	1.80	0.52	37.30
2	60.67	3.88	0.85	0.52	34.08

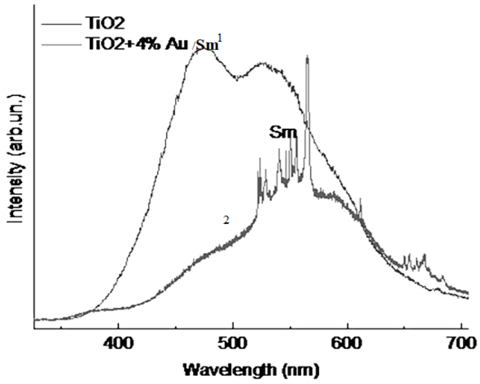
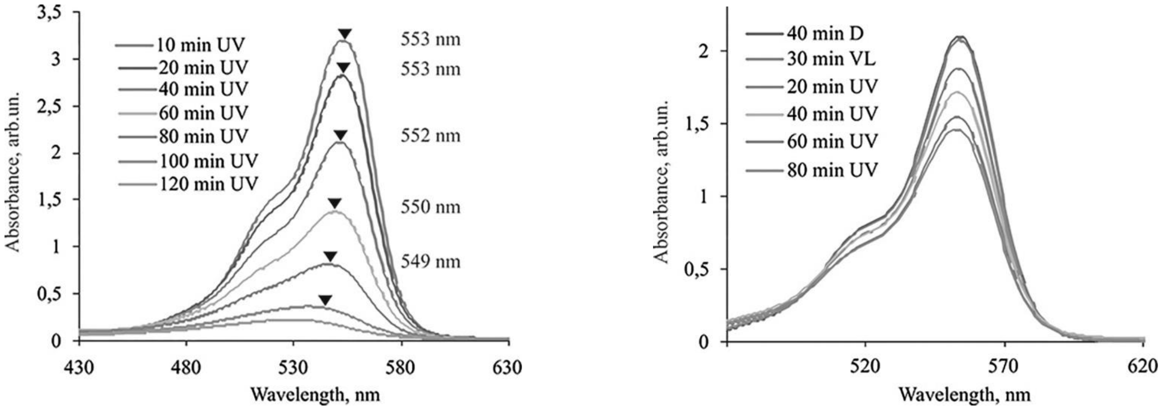


Fig. 4. PL spectra of TiO_2 (1) and TiO_2&Au/Sm (2)

Photocatalytic activity of the TiO_2&Au/Sm nanopowders. The analysis of the UV-VL absorbance spectra shows that the TiO_2&Au/Sm nanocomposite particles exhibit high sorption and photocatalytic activity in the destruction of cationic dyes – MB and RhB. In particular, due to the adsorption on TiO_2&Au/Sm particles ([Au] = 2 wt. %) MB initial concentration decreases from 10 to 5.75 mg/dm^3, for RhB under the same conditions to 5.81 mg/dm^3, and RhB from 20 mg/dm^3 to 15.8 mg/dm^3. At the same time, for TiO_2&Au/Sm ([Au] = 4 wt. %) the MB concentration decreases from 10 to 1.02 mg/dm^3 in 5 min contact. For anionic dyes – OG and MO, the sorption activity is significantly lower. The concentration of MO in the presence of

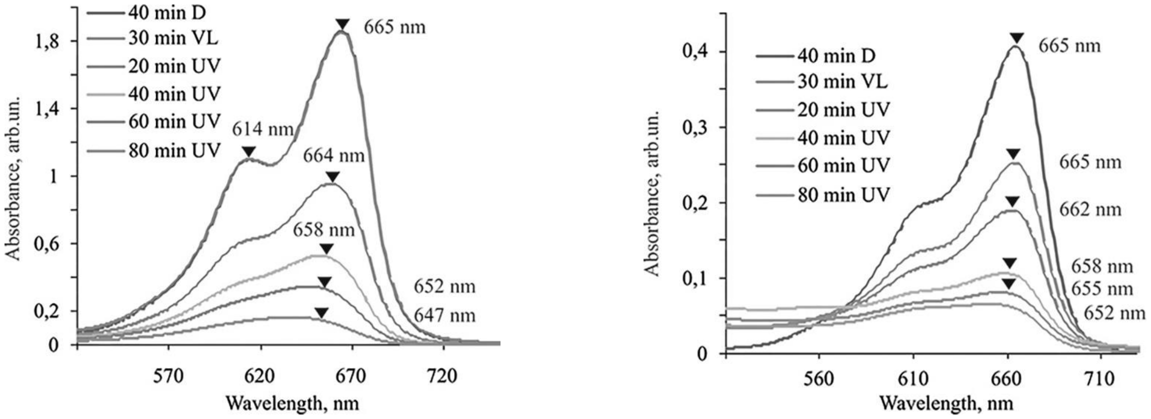
TiO_2&Au/Sm particles ([Au] = 2 wt. %) decreases from 20 to 15.4 mg/dm^3. In the presence of TiO_2&Au/Sm particles ([Au] = 4 wt. %), it decreases from 10 to only 8.7 mg/dm^3, and the concentration of OG lowers from 10 to 6.5 mg/dm^3, respectively.

The UV-VL spectra of RhB and MB solutions obtained after their UV irradiation in the presence of TiO_2&Au/Sm composites are presented in Figs. 5–6. The degradation degree of the solutions after 20 and 80 min of UV light is summarized in Table 3.



a *b*

Fig. 5. UV-VL spectra of RhB solutions obtained in the presence of TiO_2&Au/Sm, where [Au] wt. % is: *a* – 2, *b* – 4



a *b*

Fig. 6. UV-VL spectra of MB solutions obtained in the presence of TiO_2&Au/Sm where [Au] wt. % is: *a* – 2, *b* – 4

Table 3. The degradation degree of the dye after contact with TiO_2&Au/Sm powder under UV light

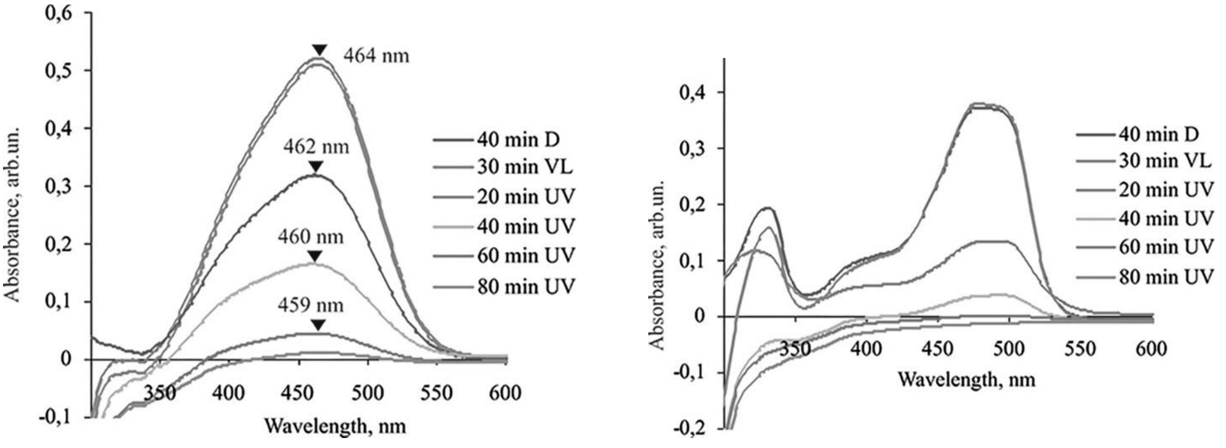
UV, min	TiO_2&Au/Sm ([Au] 2 wt. %		TiO_2&Au/Sm ([Au] 4 wt. %	
	RhB	**MB**	**RhB**	**MB**
20	84.0	81.5	10.5	95.0
80	98.5	96.0	24.1	98.6
	MO	**OG**	**MO**	**OG**
20	63.1	36.5	15.5	28.3
80	89.2	46.7	22.6	41.5

So, the degradation of the MB solution reaches 96.0–98.6 %. Such a process is accompanied by the rapid disappearance of the peak at 614 nm and the hypsochromic shift of the maximum from 665 to 652 nm, which indicates the destruction of dye molecules with the formation of intermediate products. A hypsochromic shift of the maximum from 553 to

549 nm is also observed for the RhB spectrum. Besides, particles with a lower Au content exhibit photocatalytic activity during the degradation of MO and OG solutions (Fig. 7, Table 3). For MO spectra, a shift of the maximum from 464 to 459 nm is also observed.

Summarizing the results of the study according to the degree degradation of dye solutions with TiO_2&Au/Sm particles, the efficiency ranges were determined: OG < MO < MB < RhB, for TiO_2&Au/Sm ([Au] = 2 wt. %), MO < RhB < OG < MB, for TiO_2&Au/Sm ([Au] = 4 wt. %).

The difference in the effectiveness of the degradation of the solutions can be related to the PZC of the composite nanoparticles, which corresponds to pH values of 7.01 ([Au] = 2 wt. %) and 9.25 ([Au] = 4 wt. %) (Table 1).



a *b*

Fig. 7. UV-VL spectra of: *a* – MO, *b* – OG solutions in the presence of TiO_2&Au/Sm particles, where [Au] is 2 wt. %

Thus, the results are shown in Fig. 5–7 in the form of UV-VL absorption spectra in the range of 200–800 nm and include the full absorption spectrum of the dye, consisting of chromophoric and aromatic parts. The chromophoric component is characterized by absorption peaks in the range of 500–720 nm. It characterizes the double bonds of groups during the irradiation of the dye with subsequent loss of color of its solution. At the same time, in the range of 230–280 nm, there are absorption peaks for the aromatic component of the dye, due to its destruction, and complete mineralization to CO_2 and H_2O formation. The degree of decomposition of the dye can be regulated by the concentration of the dye, as well as the concentration of nanoparticles of the photocatalyst. An important role is also play the electrokinetic properties of nanoparticles, which can be the subject of a separate study. The authors present the PZC of the synthesized TiO_2&Au/Sm^{3+} nanocomposites.

CONCLUSION

TiO_2&Au/Sm nanocomposites with samarium ions of 0.2 wt. % and gold ions of 2 and 4 wt. % were obtained by the method of chemical deposition of TTIP with solutions of samarium nitrate and gold hydrochloric acid followed by heat treatment of the precipitates. The CSR of the particles is correspondingly 18 and 20 nm. The inclusion of Au cations in the structure leads to an increase in the parameters of the anatase crystal lattice. The inclusion of Sm in the structure of the sample was proved by EDS and PL methods. Nanocomposite shows low photoluminescence, and high sorption activity to cationic dyes (MB and RhB) and slightly for anionic ones (MO and OG). Degradation of dye’s under UV light is accompanied by a hypsochromic shift of the characteristic maxima, which indicates the destruction of dye molecules with the formation of intermediate products. The degree of dye degradation in the presence of the TiO_2&Au/Sm particles ([Au] 2 wt. %), is higher than in the presence of the TiO_2&Au/Sm particles ([Au] 4 wt. %), except the MB solution.

ACKNOWLEDGEMENT

Dr O.M. Lavrynenko is supported by the PAUSE program, a national emergency program for scientists and artists in exile, run by the Collège de France. We express gratitude to O.F. Kolomys (V. Lashkaryov Institute of Semiconductor Physics NAS of Ukraine) for the PL spectra.

Структура, фотолюмінесценція та фотокаталітична активність нанокомпозитів TiO_2@Au/Sm^{3+} для ремедіації барвників

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Текстильна промисловість виробляє багато токсичних барвників і пігментів, які можуть поглинати та відбивати сонячне світло у воді. Останнім часом застосування гетерогенного фотокаталізу для видалення цих забруднювачів є дуже ефективним з використанням нанокомпозитів на основі оксиду металу з благородними та рідкісноземельними металами під різним світловим опроміненням. Вплив концентрації допанту на структурні, оптичні та електронні властивості наноматеріалів TiO_2 також важливий для їхньої фотокаталітичної ефективності. Синтез частинок TiO_2&Au/Sm із вмістом золота 2 і 4 мас. % та самарію 0.2 мас. % здійснювали шляхом хімічного осадження розчину ТТІП (титан тетраізопропоксиду) в присутності катіонів Sm^{3+} та Au^{3+}. ОКР нанопорошків становив 18–20 нм. Показано, що в структуру композиту входить золото зі збільшенням параметрів кристалічної ґратки анатазу. Наявність самарію в структурі композиту підтверджена методами ЕДС та ФЛ. Отримані структури виявили високу сорбційну активність до катіонних барвників (метиленового блакитного та родаміну Б), меншу – до аніонних, зокрема, Orange G (помаранчевий Ж). Показано, що під впливом УФ-опромінення розчини зазнають зміни кольору, що супроводжується руйнуванням молекул барвника, яке підтверджується гіпсохромним зсувом характерного максимуму в оптичних спектрах поглинання. Серія ОЖ (46.7 %) < МП (89.2 %) < МБ (96.0 %) < Род Б (98.5 %) для частинок TiO_2&Au/Sm ([Au] = 2 мас. %, [Sm] = 0.2 мас. %) і МП (22.6 %) < Род Б (24.1 %) < ОЖ (41.5 %) < МБ (98.6 %) для частинок TiO_2&Au/Sm ([Au] = 4 мас. %, [Sm] = 0.2 мас. %) до інтенсивності знебарвлення розчинів барвника. Різниця в перебігу фотокаталітичного процесу може бути пов’язана з ТНЗ (точка нульового заряду) композитних частинок, яка становить 7.01 ([Au] = 2 мас. %) і 9.25 ([Au] = 4 мас. %).

***Ключові слова**: титан тетраізопропоксид, анатаз, самарій, золото, органічні барвники, УФ опромінювання, фотокаталіз*

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Received 09.08.2024, accepted 05.03.2025