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Morphology, surface structure, and photoluminescence of nanosized titania composites with oxide carbonitride (C₃N₄O_x) for optic application

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The morphology and structure of the prepared nanomaterials were characterized by scanning electron microscopy, infrared, soft X-ray emission spectroscopy (SXES C K α), X-ray photoelectron spectroscopy (XPS), and photoluminescence (PL). The combination of C₃N₄O_x with anatase in the composite allowed the expansion of the photosensitivity of TiO₂ in the visible spectrum region, with spectral absorbance occurring above 400 nm and decreasing band gap to 2.2 eV.

Keywords: Optical activity, SXES C K α , XPS, photoluminescence, C₃N₄O_x/TiO₂.

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Introduction

It is known that carbon nitride (g-C₃N₄) is a precursor for metal and non-metal nanoparticles, which enhances the photocatalytic activity and selectivity of photocatalysts [1-8] due to its graphite-like structure and energy band gap.

Authors [5] have established that the energy band gap of graphitic g-C₃N₄ is ~ 2.7 eV (460 nm) with a perfect electronic band structure (CB and VB edge positions at approx. -1.1 V and + 1.6 V vs NHE (normal hydrogen electrode); therefore, g-C₃N₄ is a semiconductor with thermal stability because of the strong covalent bond between C and N atoms.

The creation of an active composite material based on C₃N₄ is an urgent task. A number of studies [7-11] have shown that the synergistic effect of the combination of g-C₃N₄ [12] or melamine [13] and TiO₂ lead to improved optical activity. For example, the g-C₃N₄/TiO₂ nanocomposite was obtained by thermal decomposition of urea on TiO₂ (P25) [10]. The average pore diameter of the nanostructures was found to be 3.7 nm. The specific surface areas of g-C₃N₄/TiO₂ and g-C₃N₄ were found to

be 29.4 and 36.2 m²/g, respectively. The band gap values (E_g) were 2.70 and 2.75 eV, significantly lower than those of P25. Unfortunately, the g-C₃N₄/TiO₂ composite, as well as g-C₃N₄, has limited use of solar energy, because they are active only in the near-blue region of the visible spectrum (wavelengths of less than 460 nm). So, it is proposed to combine C₃N₄O_x with TiO₂ in the anatase phase for the best heterojunction with electron transactions, followed by intensive PL and E_g reducing.

The aim of the work was to study the effect of C₃N₄O_x on the electronic states of O, Ti, and H atoms, on the surface of titanium dioxide, the structure and photoluminescence of the obtained nanocomposites.

I. Methodology

Materials. All reagents and solvents were purchased from commercial sources. Anatase powder (10-20 nm) was produced strictly as reported in Ref. [9]. Due to the heat treatment of melamine for 0.5, 1.0, and 1.5 h in the highest temperature reaction zone at 580°C, in a ceramic crucible with anatase powder, the as prepared

nanocomposites were formed and labeled as $C_3N_4O_x/TiO_2$ -0.5 h, $C_3N_4O_x/TiO_2$ -1.0 h, and $C_3N_4O_x/TiO_2$ -1.5 h, respectively [14].

Instrumentation. High-resolution electron microscopy and EDS were measured with a MIRA 3 TESCAN instrument. Soft X-ray emission spectroscopy (SXES) and X-ray photoelectron spectroscopy (XPS) were employed in the present work to study the electronic structure and chemical surface states of the $C_3N_4O_x/TiO_2$ (anatase)-1.5 h and $C_3N_4O_x/TiO_2$ (rutile)-1.5 h samples. The SXES C K α bands, giving information on the energy distribution of C 2p electronic states, have been studied following the technique reported in ref. [15]. In brief, the SXES C K α bands were measured with an energy resolution of about 0.2 eV, employing an RSM-500 spectrometer-monochromator possessing a diffraction grating with 600 grooves per 1 mm and an $R = 6026$ mm radius of curvature. Operating conditions of a spectrometer electron gun were set to be following: accelerating voltage 5.2 kV, anode current 3.6 mA. Employing these operational conditions of the spectrometer electron gun, we did not detect any changes in the shape of the SXES C K α bands during spectra acquisition. The XPS spectra were measured with a UHV-Analysis System designed and assembled by SPECS Surface Nano Analysis Company (Germany), following the technique reported elsewhere [16]. All UV-visible spectroscopic measurements were made using a Perkin Elmer Lambda Bio 35 UV-Vis

spectrometer allowing a band gap analysis [14]. IR spectra were recorded using the method described elsewhere [17].

II. Results and Discussion

SEM

The study of SEM morphology presents that $C_3N_4O_x$ particles possesses a plate-like shape with pores mainly of 15-30 nm in size (Fig. 1 a-d). As a result of such a structure, defects with active centers can form on the surface due to the presence of oxygen atoms. SEM results show the similarity of the crystal structure of $C_3N_4O_x$ with graphite itself. SEM micrographs of $C_3N_4O_x/TiO_2$ (anatase) composite nanoparticles demonstrate their arrangement in the form of separate globular NPs and clusters of TiO_2 between the plates and in the channels of the scaly porous $C_3N_4O_x$ plates (Fig. 2 a-b).

IR spectra

The analysis of IR spectra shows that initial samples of titanium oxide are characterized by absorption bands of Ti-O oscillations in the region $\nu \sim 1080\div450$ cm^{-1} , with frequencies $\nu \sim 518$ and 670 cm^{-1} for rutile [18], and $\nu \sim 504$ and 664 cm^{-1} for anatase (Fig. 3). For anatase (Fig. 3, curve 1), there is a region of absorption peaks at $3350\text{--}3250$ cm^{-1} , 1605 cm^{-1} , the band of water molecule

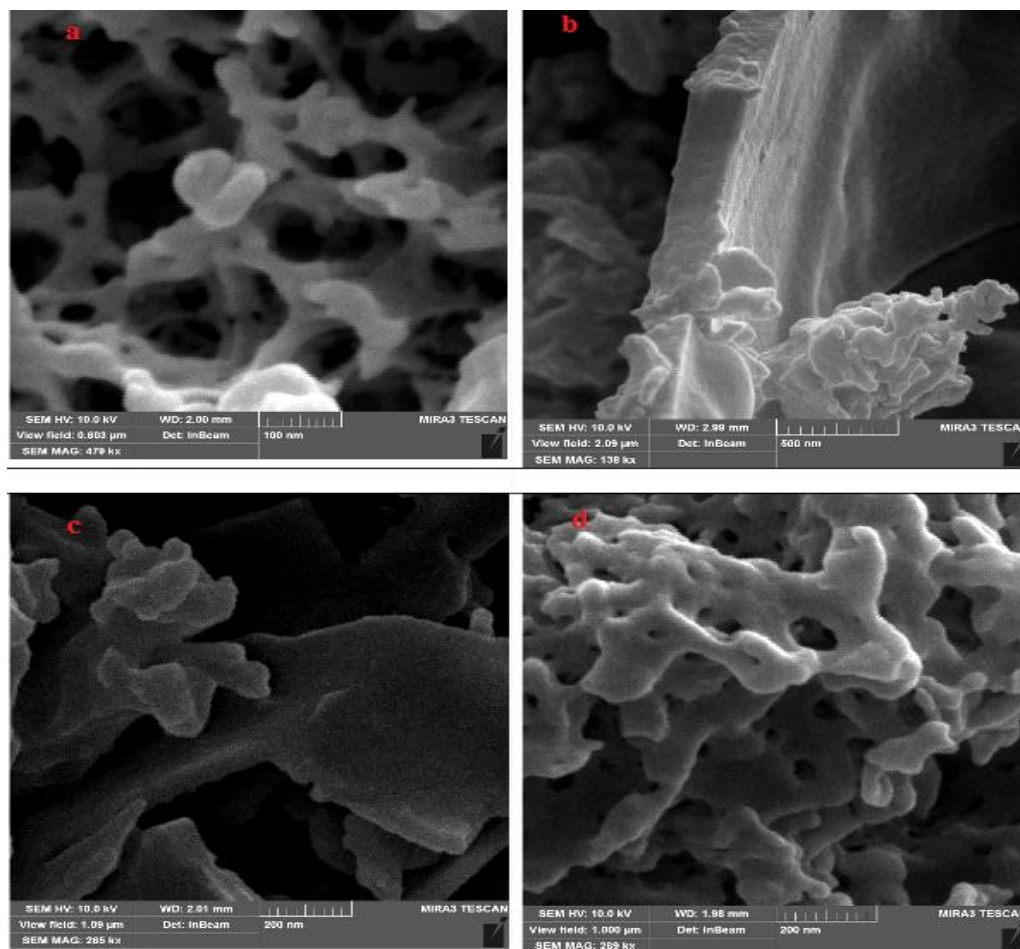


Fig. 1. Morphology of $C_3N_4O_x$ from melamine:
a, d pores, b – side view of the plate, c – plates in a horizontal position.

deformation vibrations, as previously was detected in [19]. Authors [19] have noted that water adsorption on TiO_2 surface originates due to the presence of OH groups and stronger electron-acceptor centers Ti^{4+} . The pristine $\text{C}_3\text{N}_4\text{O}_x$ sample is characterized by a set of absorption bands (C–N, C=N, C=C, C≡N, C–O, C–NH₂, C=NH, OH), shown in Fig. 3 by curve 2. The absorption band with a frequency of ν 810 cm^{-1} corresponds to vibrations of the radial mode of heptazine rings, as in crystalline potassium poly (heptazine imide) of Ref. [20]. The IR absorption spectrum of the composite is characterized by a total set of absorption bands of the original rutile and carbon nitride, with a change in the position of frequencies, intensity, and the disappearance of some bands and the appearance of new ones [14].

The absorption bands at frequencies $\nu \sim 984$ cm^{-1} , $\nu \sim 2172$ cm^{-1} disappear, and shoulders appear at frequencies $\nu \sim 1583$ cm^{-1} and $\nu \sim 3237$ cm^{-1} , as well as an absorption band at $\nu \sim 3086$ cm^{-1} (see Fig. 3 A, curve 3). The intensity of the absorption bands in the frequency range $\nu \sim 3500 \div 2900$ cm^{-1} weakens. Additionally, oxygen doping of carbon nitride leads to a decrease in the intensity of the characteristic absorption bands in the

range of $1200 \div 1650$ cm^{-1} compared to the structure of oxygen-free g- C_3N_4 and $\text{C}_3\text{N}_4/\text{TiO}_2$ (rutile) [18].

SXES C K α and XPS

Fig. 4 presents the SXES C K α bands as measured for the $\text{C}_3\text{N}_4\text{O}_x/\text{TiO}_2$ (rutile)-1.5 h and $\text{C}_3\text{N}_4\text{O}_x/\text{TiO}_2$ (anatase)-1.5 h samples under study. For comparison, the same graphite band is also plotted in this figure. As can be seen from this figure, the shapes of the SXES C K α bands of these samples resemble that of graphite. These concern a number of the fine-structure peculiarities (a to e), as well as their energy positions. Most importantly, the SXES C K α bands of the samples feature the well-resolved peculiarity “e” (the so-called π -sub-band), which is characteristic for graphite and other carbon-containing materials with sp^2 -hybridization [15, 21].

The existence of sp^2 -hybridization for carbon, presented in both samples, is confirmed by the XPS measurements (Fig. 5). As can be seen from Fig. 5a, the C 1s core-level spectra reveal a shoulder at binding energies around 293 eV, which is characteristic for carbon being in sp^2 -hybridization state [22]. Further, the

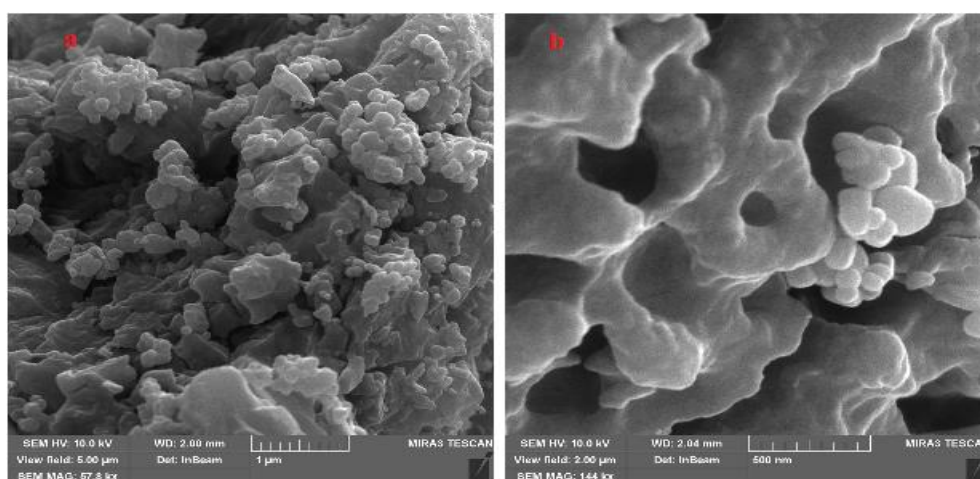


Fig. 2. Morphology of nanocomposite $\text{C}_3\text{N}_4\text{O}_x/\text{TiO}_2$ -1.5 h: a – magnification 57, 800 (general view of TiO_2 particles on the $\text{C}_3\text{N}_4\text{O}_x$ surface); b – magnification 144,000 (clusters of TiO_2 nanoparticles in $\text{C}_3\text{N}_4\text{O}_x$ channels).

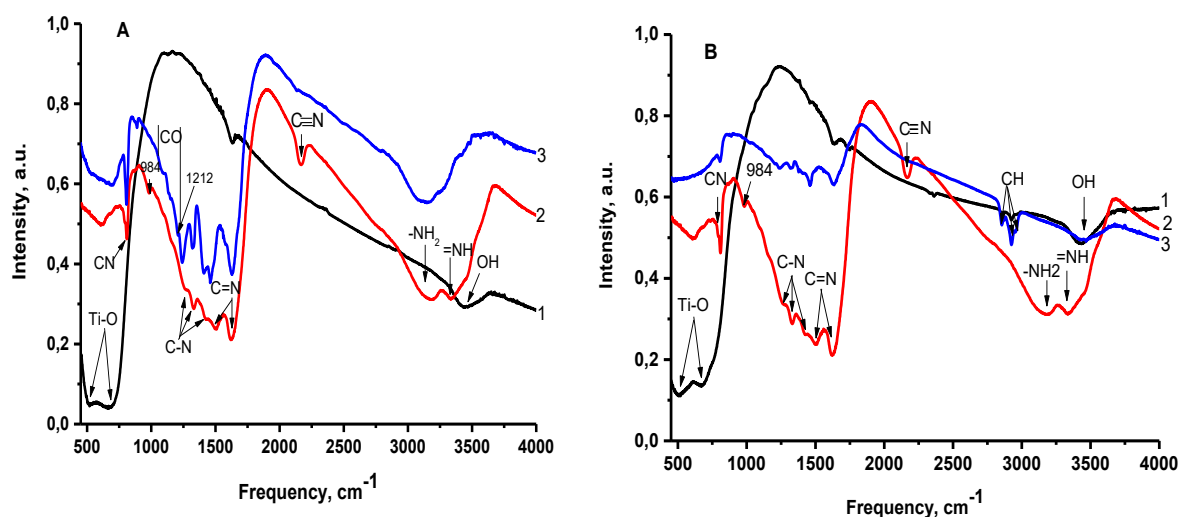


Fig. 3. IR spectra of nanosystems with TiO_2 : **A** 1 – TiO_2 rutile, 2 – $\text{C}_3\text{N}_4\text{O}_x$, 3 – TiO_2 (rutile)/ $\text{C}_3\text{N}_4\text{O}_x$; **B** 1 – TiO_2 anatase, 2 – $\text{C}_3\text{N}_4\text{O}_x$, 3 – TiO_2 (anatase)/ $\text{C}_3\text{N}_4\text{O}_x$.

maximum of the C 1s spectrum, located at about 287.8 ± 0.1 eV, can be formed by $\text{C}=\text{O}$ bonds [23], while peculiarity at about 284.5 ± 0.1 eV is obviously due to hydrocarbons adsorbed on the sample surface as a result of its contact with laboratory air, whereas small shoulder nearly 281.8 eV can be formed by Ti–C bonds [24]. This binding energy is characteristic for titanium being in the charge state +4, whereas the binding energy of the XPS O 1s spectra (529.8 ± 0.1 eV) corresponds to oxygen involved in the Ti–O bonds of TiO_2 [14, 24]. Detailed study of the XPS N 1s core-level spectra (Fig. 5b) indicates that their maxima are at about 398.7 ± 0.1 eV, being close to those detected in transition metal nitrides [24].

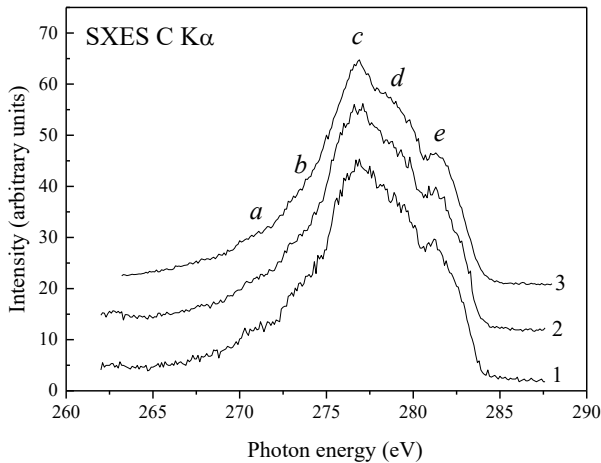


Fig. 4. SXES C K α bands of (1) $\text{C}_3\text{N}_4\text{O}_x/\text{TiO}_2(\text{rutile})$ -1.5 h, (2) $\text{C}_3\text{N}_4\text{O}_x/\text{TiO}_2(\text{anatase})$ -1.5 h, and (3) graphite.

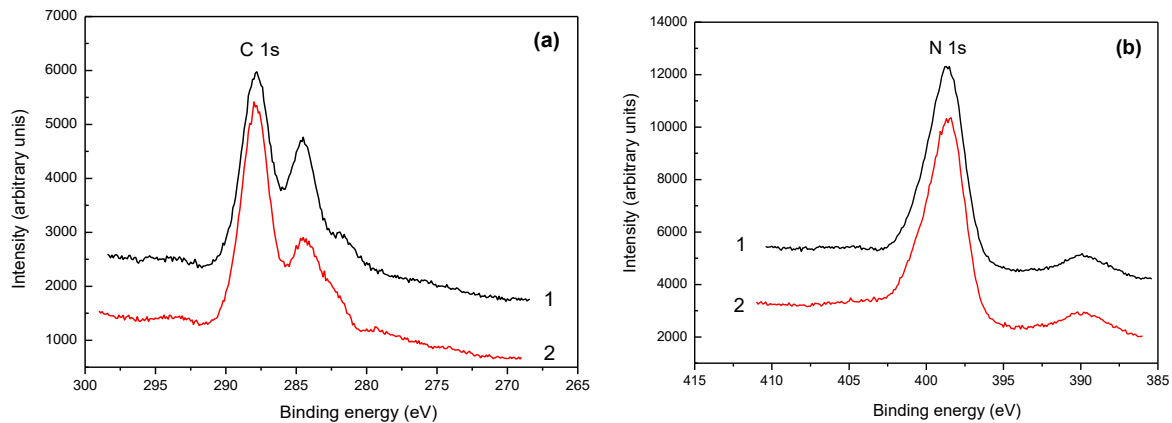


Fig. 5. XPS spectra of (1) $\text{C}_3\text{N}_4\text{O}_x/\text{TiO}_2(\text{rutile})$ -1.5 h and (2) $\text{C}_3\text{N}_4\text{O}_x/\text{TiO}_2(\text{anatase})$ -1.5 h: (a) C 1s and (b) N 1s.

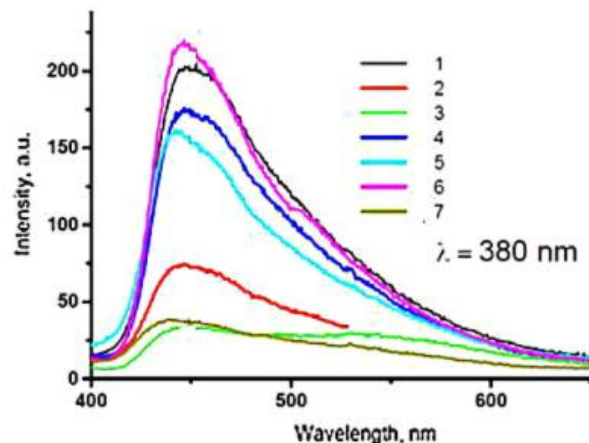


Fig. 6. PL spectra of nanocomposites $\text{C}_3\text{N}_4\text{O}_x/\text{TiO}_2$ at $\lambda_{\text{exc}} = 380$ nm.

PL spectra

The photoluminescence spectra of the $\text{C}_3\text{N}_4\text{O}_x$ and $\text{C}_3\text{N}_4\text{O}_x/\text{TiO}_2(\text{anatase})$ samples were studied under 380 nm light excitation (Fig. 6) compared to [26–29]. The samples synthesized with a thermal treatment of 1.5 h at temperatures of 540, 560, and 580 °C were assigned as curves 1, 2, and 3. Curve 4 corresponds to the $\text{C}_3\text{N}_4\text{O}_x$ sample. Curves 5, 6 and 7 correspond to the $\text{C}_3\text{N}_4\text{O}_x/\text{TiO}_2(\text{anatase})$ samples obtained with a thermal treatment over 1.0 h at temperatures of 540, 560, and 580 °C. Moreover, we observed an emission peak at about 450 nm in the visible spectral region for $\text{C}_3\text{N}_4\text{O}_x$ and $\text{C}_3\text{N}_4\text{O}_x/\text{TiO}_2$. Comparison of the photoluminescence bands of the obtained samples when excited by light with the $\lambda_{\text{exc}} = 380$ nm shows that the shape of the photoluminescence bands and the positions of their maxima are close to those detected previously in Refs. [26–28] at 350 nm for C_3N_4 and $\text{C}_3\text{N}_4/\text{TiO}_2$; a significant decrease in the intensity of the photoluminescence bands of the samples is observed with increasing the temperature of their synthesis.

Thus, the XPS and IR surface analysis confirmed the presence of hydroxyl groups and oxygen vacancies with successful interfacial interactions in graphite-like $\text{C}_3\text{N}_4\text{O}_x/\text{TiO}_2(\text{anatase})$ -1.5 h (580 °C) due to its porous structure. Additionally, oxygen doping of carbon nitride results in a reduction in the intensity of the characteristic absorption bands within the $1200\text{--}1650\text{ cm}^{-1}$ range (Fig. 3B) for the $\text{C}_3\text{N}_4\text{O}_x/\text{TiO}_2$ composite compared to oxygen-free g- C_3N_4 . Furthermore, effective active centers of N, oxygen vacancies and Ti^{4+} are confirmed by Ti–N and Ti–O bonds. $\text{C}_3\text{N}_4\text{O}_x$ exhibited an intense, broad peak centered at approximately 450 nm, which can

be explained by the band-to-band PL phenomenon, where the energy of the light is equal to the band gap energy of C_3N_4 [26-28]. The effective charge separation occurs between $C_3N_4O_x$ and TiO_2 , whereby photogenerated holes in TiO_2 are transferred from the valence band (VB) to the highest occupied molecular orbital (HOMO) of $C_3N_4O_x$, followed by the injection of electrons from the lowest unoccupied molecular orbital (LUMO). The increased content of O in $C_3N_4O_x$ compared to TiO_2 is caused by the presence of O_2 during the possible exfoliation process (according to XPS results, the O 1s spectra have two characteristic peaks at binding energies of 531.2 and 529.4 eV, respectively; these correspond to oxygen belonging to C-O bonds in $C_3N_4O_x$ and Ti-O bonds in the anatase phase). We can conclude that the charge separation in this system may occur via a type-II heterojunction mechanism based on the obtained results and references [26-28]. Upon light irradiation, both semiconductors enter an excited state, followed by the transfer of photogenerated electrons from the CB of $C_3N_4O_x$ to the TiO_2 CB (conduction band). The TiO_2 band (VB) is at a lower energy level than that of graphitic-like $C_3N_4O_x$, so holes (h^+) can migrate from TiO_2 to $C_3N_4O_x$. A Z-scheme heterojunction is also possible, wherein photogenerated electrons from the TiO_2 CB rapidly recombine with holes from the $C_3N_4O_x$ VB. Consequently, active electrons can accumulate in the $C_3N_4O_x$ CB, while oxidizing holes accumulate in the TiO_2 VB. The presence of atomic oxygen (O_x) can enhance the internal electric field at the interface, thereby accelerating interfacial charge carriers transfer and reducing both photoluminescence (PL) intensity and the energy gap.

Conclusions

The $C_3N_4O_x/TiO_2$ nanocomposites were synthesized by pyrolysis of melamine at temperatures between

540÷580°C. To develop the new semiconductor nanocomposite, $C_3N_4O_x$ was combined with TiO_2 nanoparticles via chemical vapor deposition (CVD), leveraging surface defects. IR spectroscopy and XPS analysis revealed the deposition of the melamine precursor on the TiO_2 nanoparticles, and the interaction occurring between the titanium dioxide and the C and N atoms of carbonitride oxide (Ti-O, C-N, C=O, and Ti-C bonds are present). The $C_3N_4O_x$ (carbon nitride oxide)/ TiO_2 nanocomposite was found to have higher photosensitivity than anatase due to several of advantages, including reduced photoluminescence with a band gap of 2.2 eV, and more efficient separation of electron-hole pairs. SXES C K α and XPS measurements confirmed the existence of sp^2 -hybridization for carbon in the samples with anatase and rutile matrices.

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Conflicts of Interest. The authors have no conflicts of interest to declare.

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Морфологія, поверхнева структура і фотолюмінесценція нанорозмірних композитів на основі діоксиду титану з оксидним карбонітридом (C₃N₄O_x) для застосування в оптиці

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Морфологія та структура отриманих наноматеріалів були охарактеризовані за допомогою скануючої електронної мікроскопії, інфрачервоного випромінювання, м'якої рентгенівської емісійної спектроскопії (МРЕС С К α), рентгенівської фотоелектронної спектроскопії (РФЕС) та спектрів фотолюмінесценції (ФЛ). Поєднання C₃N₄O_x з анатазом у композиті дозволило розширити fotocутливість TiO₂ у видимій області спектра, зі спектральним поглинанням понад 400 нм та звуженням енергетичної щільності до 2,2 еВ.

Ключові слова: Оптична активність, МРЕС С К α , РФЕС, фотолюмінесценція, C₃N₄O_x/TiO₂.