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PHOTOCATALYSIS EFFECT-BASED OPTICAL INFORMATION RECORDING ON THE TITANIUM DIOXIDE NANOPARTICLES AND LUMINESCENT DYE-DOPED POLYMER NANOCOMPOSITE

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Abstract. Titanium dioxide is the most widely used semiconductor substance as a photocatalytic material in self-cleaning surfaces, air and water purification systems, sterilization, hydrogen evolution, and photoelectronchemical conversion. In this work, we propose a photocatalysis effect-based optical information recording on the titanium dioxide nanoparticles and luminescent dye-doped polymer nanocomposite for the first time. The optical information was recorded as holographic gratings. Holographic and non-holographic methods were employed to record optical information on a polymer nanocomposite. A green laser beam and a halogen-tungsten lamp with a band-pass filter were used for this purpose. As a result, the high-density optical information was obtained, with an optical density of 500 mm^{-1}. The data recorded on the polymer nanocomposite is stable and durable.

Keywords: Titanium Dioxide, Polymer Nanocomposite, Luminescent Dye, Photocatalysis, Optical Information

1. Introduction

Similar to the structures made up of organic macromolecules,[1-5] nanoscale materials consisting of nanoparticles exhibit unique physical, chemical, and electrical properties due to their size, shape, and surface chemistry, which sets them apart from bulk materials.[6-8] Photocatalysis is a photo-activated chemical reaction that occurs when a substance encounters high-energy photons. Nanotechnology and photocatalysis are two rapidly advancing research fields in the 21st century. Several photocatalysts, such as ZnO, Fe_2O_3, CuO, and CdS, are widely used, but titanium dioxide (TiO_2) stands out for its distinctive characteristics and applications as both a semiconductor and an efficient photocatalyst. The use of photocatalytic technology has garnered significant attention due to its environmentally friendly, cost-effective, and efficient properties. In 1972, Fujishima *et al.*[9] demonstrated that TiO_2 could be used as a photoelectrocatalyst to split water into hydrogen, sparking increased research on TiO_2. In 1976, Carey *et al.*[10] utilized photocatalytic technology to treat polychlorinated biphenyls, a challenging organic pollutant, and achieved a dechlorination rate of nearly 100%. In 1977, Frank *et al.*[11] discovered that TiO_2 could effectively degrade cyanide (CN^-), marking the beginning of using photocatalytic technology for pollution control. The degradation process of photocatalytic technology can be divided into four stages: photoexcitation, carrier capture, formation of radicals, and oxidation reaction. Compared to traditional catalytic technologies, photocatalytic technology offers numerous advantages. First, the reaction conditions, such as sunlight, room temperature, and normal atmospheric pressure, are readily available and easy to attain. Second, the degradation processes and products of catalytic decomposition are pollution-free, contributing to low-carbon environmental protection. Third, the non-toxic, stable, low-cost, and recyclable characteristics further promote its development.[12] TiO_2 nanoparticles (NPs) possess versatile properties, including a high UV-absorbing effect and chemical stability, making them very advantageous materials.[13-17] This effect of photocatalysis can degrade the organic luminescent dyes (LDs). For example, Jaybhaye *et al.*[18] reported the photocatalytic degradation of organic dyes using TiO_2 and Mg-TiO_2 NPs.

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However, the biggest obstacle to applying TiO_2-based NPs is the TiO_2 high band gap energy level that requires excitation with harmful UV radiation (λ<387 nm). Doping TiO_2 with metals or non-metals usually solves this problem and shifts the absorption onset of TiO_2 to longer non-toxic wavelengths. Modifying TiO_2 with metal NPs can narrow the bandgap and increase the photocatalytic activity due to improved charge separation and reduced electron-hole recombination rates. While surface modification of TiO_2 with metal NPs results only in a small redshift of the absorption of TiO_2 NPs, decoration of the metal oxide semiconductor with organic LDs extends the absorption to a broader range of the visible light spectrum.[19] LD photosensitization means that the photosensitizer binds to the TiO_2 NPs surface by chemical or physical adsorption so that the absorption wavelength of the optical spectrum shifts to the long wavelength, thus expanding the excitation wavelength response range of TiO_2 NPs and greatly improving the utilization of visible or near-infrared parts of the optical spectrum. The mechanism of photosensitization is that once the LDs achieve their excited state by the absorption of photons in the visible range of the optical spectrum, electrons from the LD's highest occupied molecular orbital (HOMO) are transferred to their lowest unoccupied molecular orbital (LUMO) and subsequently to the conductive band (CB) of TiO_2 NPs, Fig. 1. LD used for photosensitization must meet the following characteristics: strong absorption of visible light even the part of the NIR region, photo-stability (unless the self-sensitized degradation is required), the existence of some anchoring groups (-SO_3H, -COOH, -H_2PO_3, *etc.*) and the higher excited state energy than the CB edge of TiO_2.[2]

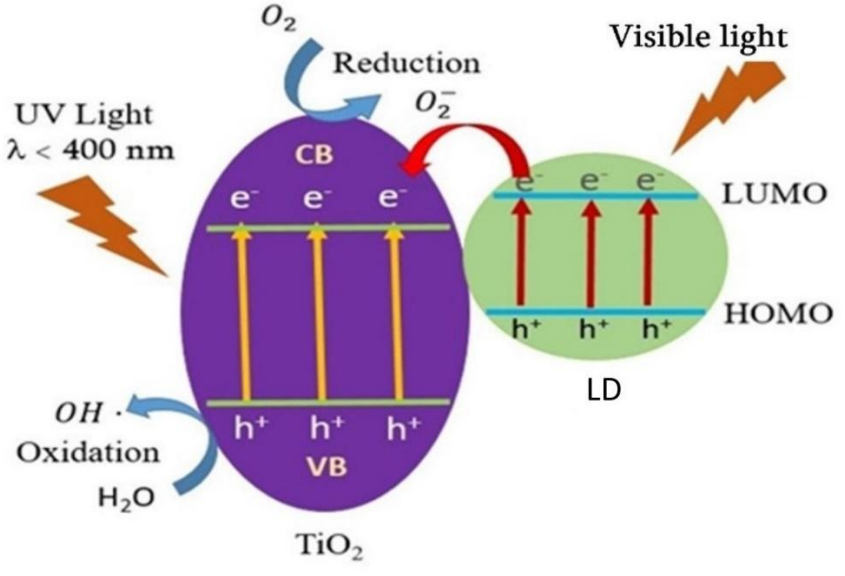

Fig. 1. Schematic representation of the photocatalytic phenomenon based on the heterojunction effect between TiO_2 NPs and LD

On the other hand, optical information storage technology involves recording information using light irradiation to change the chemical or physical state of the material in a recording medium. There are plenty of materials and methods to record high-density optical information, and the search is ongoing for methods based on the change of photo-optical characteristics of photosensitive materials, such as light absorption, reflection, scattering, refraction, polarization, and luminescence[20-24].

In this work, for the first time, we have used the effect of the photocatalytic decolorization of organic LDs to record high-density optical information as the holographic gratings using laser emission in the green range of the optical spectrum. For this purpose, we prepared a polymer nanocomposite (PNC) consisting of a polymer matrix doped with TiO2 NPs and LD Rhodamine 6G (R6G).

2. Experimental

2.1. Materials and Equipment

Macromonomer polyvinyl alcohol (PVA), with an average mol/wt 60,000-70,000 was purchased from Sigma-Aldrich. TiO_2 NPs, with 99.5% purity, and an average particle size of 70nm, were acquired from Nanografi

nanotechnology. The organic LD utilized R6G from "Exiton", Fig. 2. R6G is an inexpensive basic red dye that belongs to the xanthene class. In its powdered form, it appears reddish-violet. Typically, R6G is used as a laser dye due to its high photostability and favorable photophysical properties, including polarization and quantum yield. Additionally, it serves as a fluorescent probe for analyzing the surfaces of polymeric nano-particles, studying the structure and dynamics of micelles, and conducting single-molecule imaging in living cells. Regarding the tools used in the experiment, the absorbance and luminescence spectra of the PNC film were recorded by a computer-coupled spectrometer (Avaspec-2048, Avantes) with 1 nm accuracy. Image-based techniques, such as a fluorescence microscope (FM), a scanning electron microscope (SEM), and an atomic force microscope (AFM) were used to image the prepared nanomaterials. A digital drying chamber was utilized to obtain a uniform PNC film. Holographic gratings were recorded by exposure of the PNC film to two mutually coherent s-polarized beams of wavelength 532 nm (Nd-YAG laser) with a total exposure energy of 20mJ/cm². A He-Ne laser at 5mW intensity was used to probe for real-time and post-exposure monitoring of holographic gratings formed in the PNC film. This beam was used at a normal incidence for all experiments.

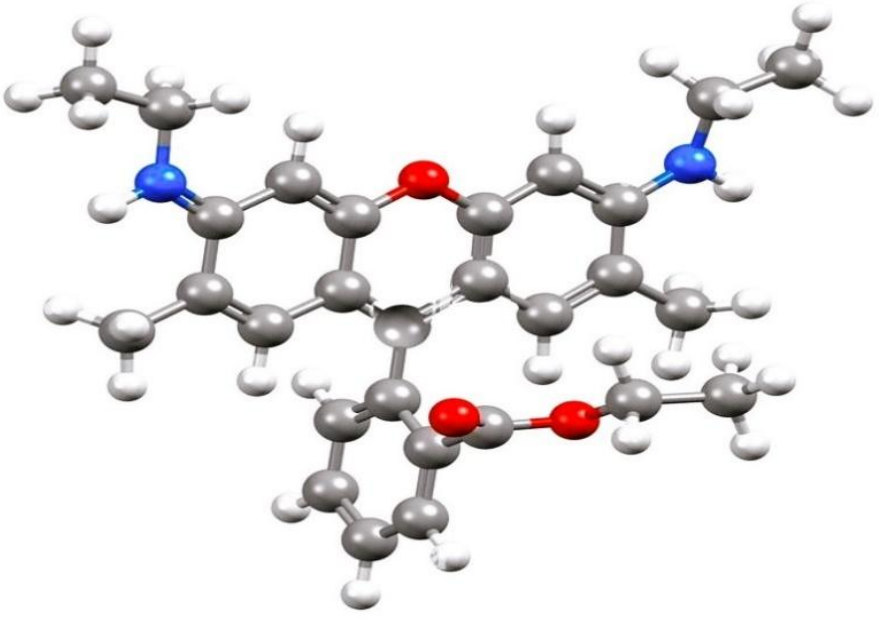

Fig. 2. The 3D structural formula of luminescence dye Rhodamin 6G

2.2. Sample Preparation

Initially, a PVA solution was prepared with the following percentage ratio in weight: 90%wt Water+10%wt PVA. To completely dissolve PVA molecules in water, the solution was heated to 80^0C and stirred at 600 rpm for 2 hours. After stirring, the solution was cooled to room temperature, and the TiO_2 NPs with average sizes of 80nm were added to obtain a nanocomposite with the following percentage ratio of constituents: 99.95 wt % (90 wt % Water+10 wt % PVA) +0.05 wt % TiO_2 NPs. The resulting solution was again stirred at 800 rpm for 45 min at room temperature. After that, a nanocomposite was filtered several times using special filters to obtain a homogeneous solution. At the final stage of preparation of the nanocomposite, the LD R6G was doped to the nanocomposite in the following percentage ratio in weight: 99.97 wt % [99.95 wt % (90 wt % Water+10 wt % PVA) +0.05 wt % TiO_2 NPs] +0.03 wt % R6G, and stirred at a speed of 600 rpm at room temperature. A drop-casting method was utilized to spread a nanocomposite homogeneously on the glass surface, which was pre-treated in deionized water. Then, a glass plate was placed in a digital drying chamber, and a nanocomposite was dried at 30 degrees for 48 hours. The resulting PNC film was carefully removed from the glass. After all procedures and solvent evacuation, a uniform PNC film with 20μm thickness was obtained, Fig. 3.

Prepared PNC film provides perfect adhesion to any surface with any curvature. Furthermore, it is environmentally friendly and distinguished by its high durability. It remains in this condition for a long period. We used an SEM to observe the distribution of TiO_2 NPs in the PNC film. Fig. 4 depicts the SEM image of the TiO2 NPs. The image reveals that the NPs are sized between 60 and 90nm and are uniformly distributed.

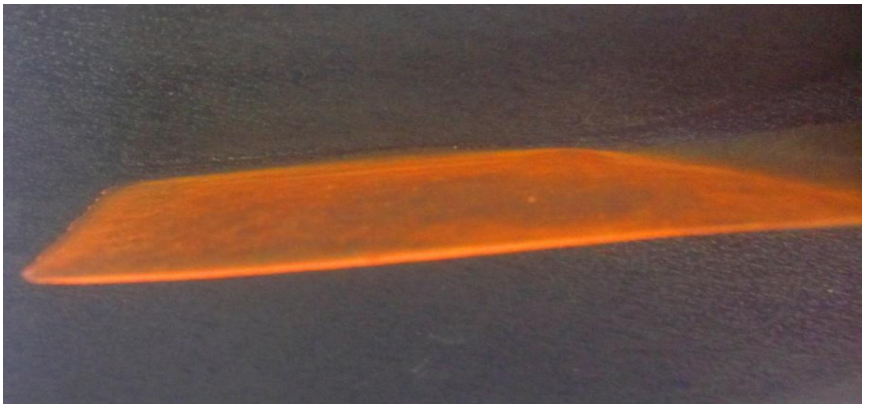

Fig. 3. Prepared PNC film

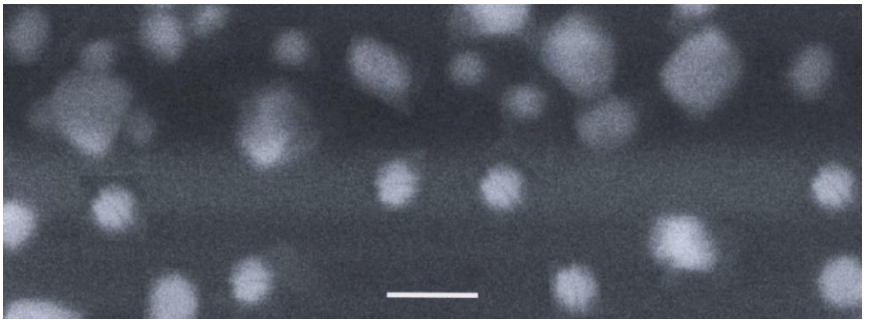

Fig. 4. SEM image of TiO_2 NPs. The scale bar is 100 nm

3. Results and Discussion

To demonstrate the degradation of LD upon exposure to visible light on the PNC film, first, we built a setup given in Fig. 5(a). The light emerging from the halogen-tungsten lamp passes through the band-pass filter, cutting the blue and red parts of the optical spectrum and permitting the green portion of light to pass. It has been shown that upon irradiation of a PNC film with green light, a rapid decomposition-degradation of R6G was caused, stimulated by the photocatalytic effect of TiO_2 NPs. In Fig. 5(b), the decomposed part appears as a dark area due to the absence of luminescence.

Using a spectrometer, the absorption and luminescence spectra of the prepared PNC film were recorded, Fig. 6.

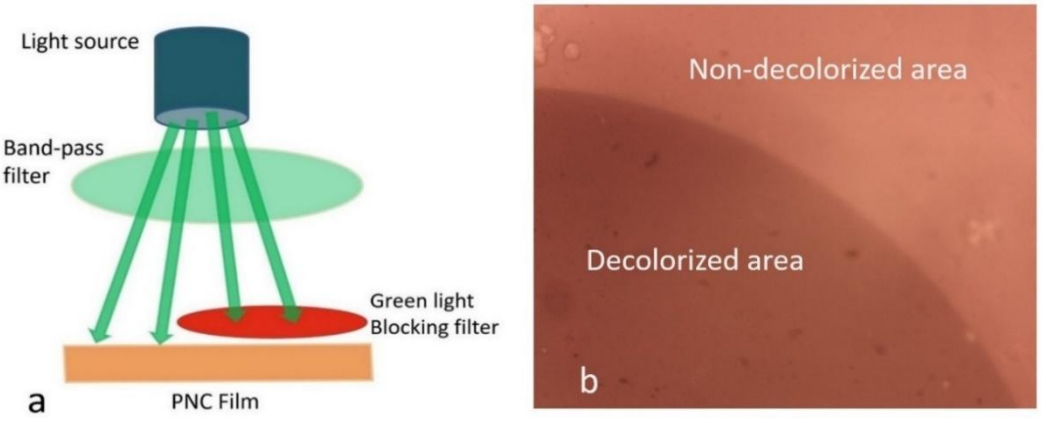

Fig. 5. Schematic setup demonstrates the LD decolorization upon exposure to green light (a). Exposed and non-exposed parts of PNC film (b)

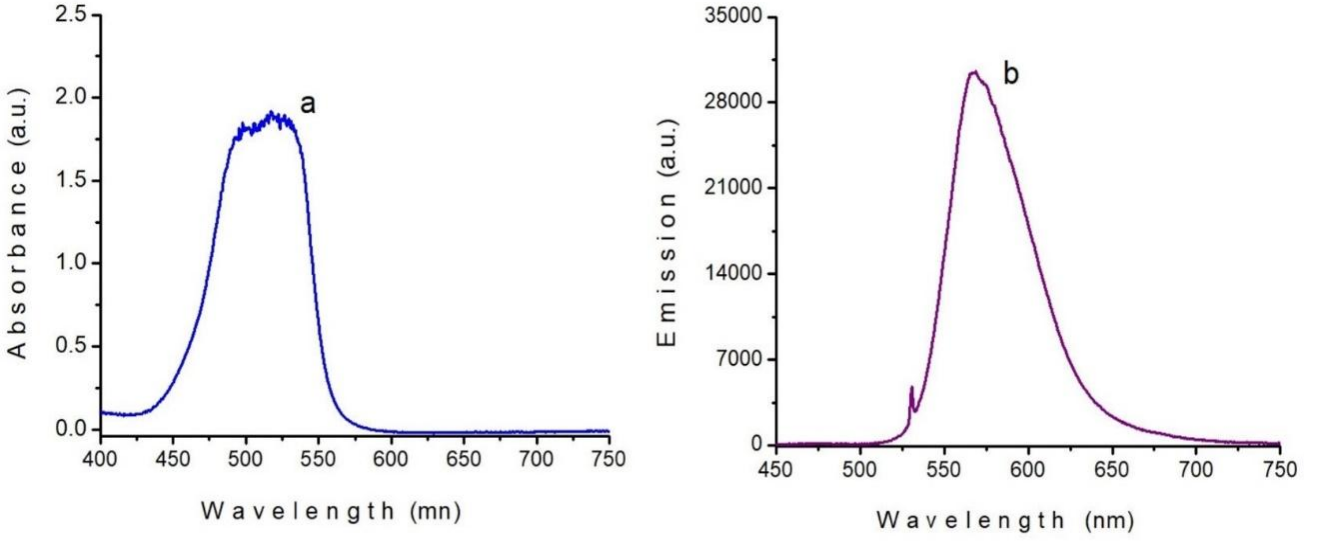

Fig. 6. Absorption (a) and luminescence (b) spectra of PNC film. The absorption maximum is located in the spectral interval of 500–530nm, while the luminescence maximum is located in the optical spectrum interval of 560–565nm

Because of its simple structure, R6G degrades easily compared to other dyes. Using photocatalytic degradation, the intensities of the absorbance and emission peaks of R6G decrease with time, and ultimately finish after a certain time, depending on the nature of the photocatalyst. The concentration of decolorized R6G is directly proportional to its absorbance and emission values. We examined the changes in R6G concentration (C/Co) linked to the variation in absorption and emission peaks during its photocatalytic decomposition.

Using a spectrometer, the photocatalytic degradation process of R6G was visualized graphically. This visualization confirms that the photocatalytic decolorization process takes place (Fig. 7).

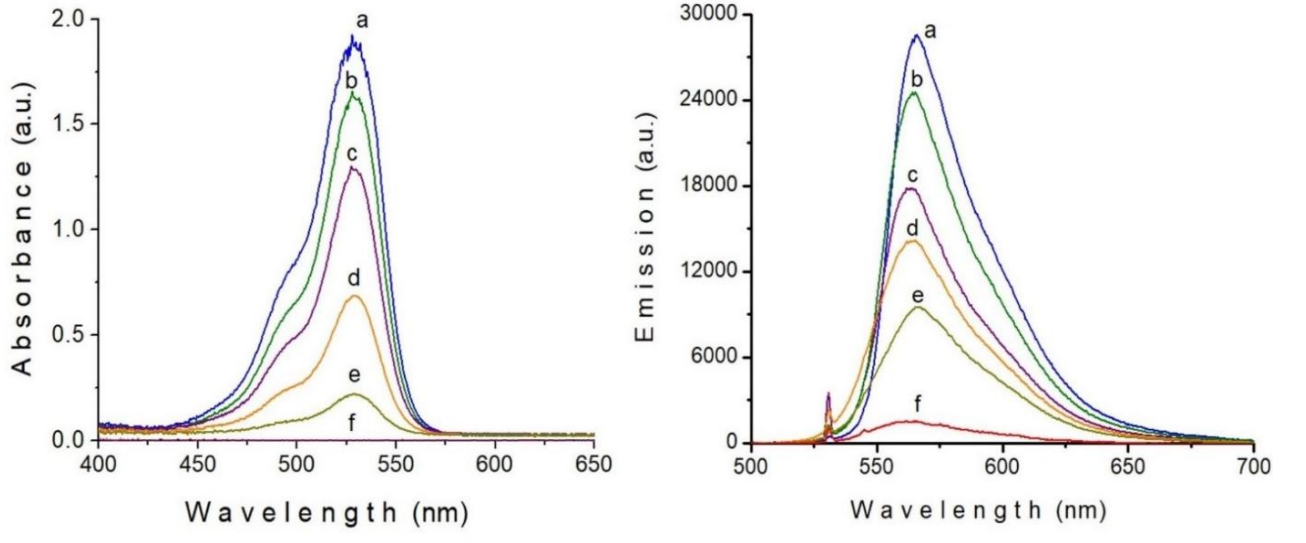

Fig. 7. Exposure-dependent changes in the absorption (left part) and luminescence spectra (right part) of an R6G upon irradiation with a laser beam. In both images, a, b, c, d, e, and f correspond to light exposures of 1, 2, 3, 4, 5, and 6 minutes

The decolorization of R6G can be explained using the following mechanism. In the initial stage, when TiO_2 NPs absorb photons of light, it triggers the generation of electron-hole pairs. This process leads to the formation of reactive species such as hydroxyl radicals ($OH^{\bullet}$), superoxide radicals ($^{\bullet}O_2^-$), and holes (h^+). These radicals act as oxidizing agents, impacting the C=N bond of the R6G molecule and breaking this double bond. Experiments

have confirmed this phenomenon, where the double bond cleavage at the nitrogen atom in azo dyes leads to the decomposition of the PNC film's irradiated parts, resulting in its photocatalytic decolorization.

The photocatalytic decolorization mechanism for R6G is shown in Fig. 8.

To record the photocatalysis effect-based optical information on the PNC film, we used the setup demonstrated in Fig. 9. The laser light beam (a) hits a half-reflective mirror (b) that reflects 50% of the total light intensity, while the other 50% passes through the half-reflective mirror and hits the PNC film. The light reflected from a mirror (b) is forwarded to a fully reflective mirror (d) rotating around the symmetry axis, from which the re-reflected beam hits the PNC film, forming a certain angle with the beam emerging from the half-reflective mirror. To provide a spatial modulation of holographic gratings ranging from 2μm to10 μm, a mirror (c) was used. As a result, the holographic diffraction gratings on the PNC with spatial frequencies ranging from 100 to 500 mm^{-1} were recorded using a CW Nd-YAG laser beam of wavelength 532 nm incident at the Bragg angle

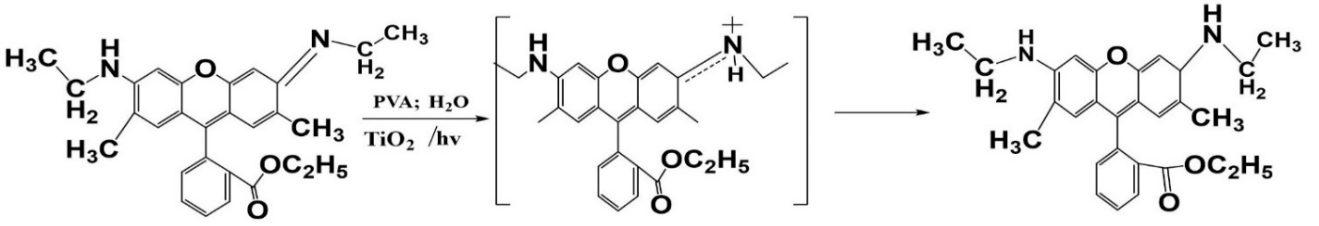

Fig. 8. The photocatalytic decolorization mechanisms of R6G

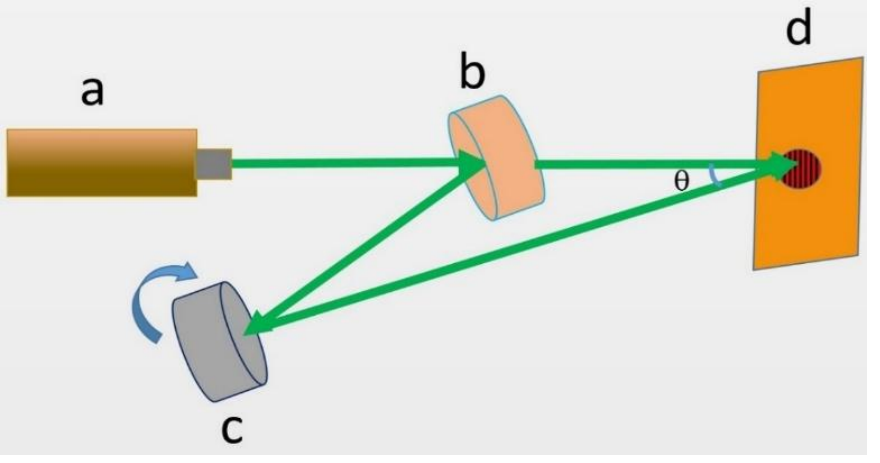

Fig. 9. Experimental setup for the recording of holographic gratings.
(a) Nd-YAG laser, (b) Half-reflective mirror, (c) Totally-reflective mirror, (d) PNC film

The recorded gratings were examined utilizing confocal and AFM microscopes. As shown in Fig. 10(a), a set of alternating bright (luminescent) and dark parallel lines, characterized by a sharp modulation of light intensities, was observed. In Fig.10(b), the AFM image of the recorded grating is demonstrated. The recorded gratings appear luminescent when illuminated with blue or green lights. It must be pointed out that besides R6G, different species of LD, such as coumarin, stilbene, and Nile red, were examined. During the experiments, we investigated and optimized such essential and significant parameters as the concentration of R6G and TiO_2 NPs on the one hand and distance-dependent energy transfer between TiO_2 NPs and R6G on the other hand.

We shone monochromatic light on a grating surface, which splits the light into specific directions due to diffraction. Each groove on the grating is a tiny slit-shaped source of diffracted light. The light diffracted by each groove combines to create a series of diffracted wavefronts. The light incident on the grating at a normal angle gives the general equation expressed as

$$d sin\theta = \lambda n$$

where n is the order of diffraction, λ is the diffracted wavelength, d is the grating constant (the distance between grooves), and θ is the angle of incidence measured from the grating normal.

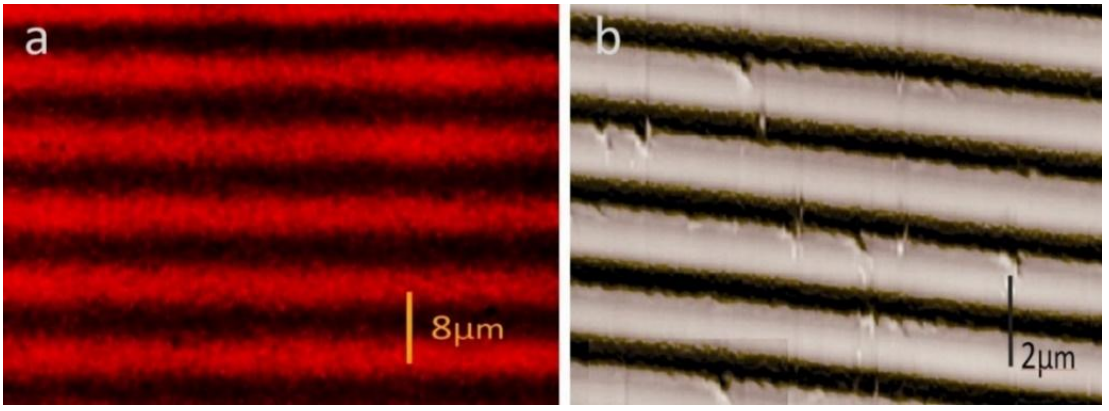

Fig. 10. Recorded gratings on the PNC pattern as it looks under a fluorescent microscope (a) and AFM (b)

Fig. 11. The He-Ne beam diffraction through the NC pattern

As seen in Fig.11, multiple diffracted orders are generated. These exhibited typical spectral and angular dependences of the first-order diffraction efficiency, with a high modulation depth and groove profiles close to sinusoidal.

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4. Conclusions

This study demonstrates, for the first time, the photocatalytic effect of high-density optical information recording using titanium dioxide nanoparticles (TiO_2 NPs) and a polymer nanocomposite doped with a luminescent dye (LD), specifically Rhodamine 6G (R6G). We prepared a polymer nanocomposite of a polyvinyl alcohol matrix infused with TiO_2 NPs and LD Rhodamine 6G. Information recording was carried out using both non-holographic and holographic methods. In the holographic process, the LD-doped polymer nanocomposite was exposed to a continuous wave (CW) Nd: YAG laser beam with a wavelength of 532 nm. This irradiation resulted in the photocatalytic decolorization of R6G, leading to the cleavage of double bonds at the nitrogen atom found in the azo dyes of R6G. Consequently, optical gratings with periods ranging from 100 to 500 mm^{-1} were successfully recorded. Experiments were also conducted using other luminescent dyes such as Coumarin, Stilbene, and Nile Red, yielding similar results. Besides, we investigated and optimized essential parameters such as the concentration of R6G and TiO_2 nanoparticles and the distance-dependent energy transfer between TiO_2 nanoparticles and R6G. The proposed polymer nanocomposite film shows great promise in developing mechanically flexible, environmentally friendly, lightweight, large-area optically recorded devices. These devices can be fabricated through room-temperature solution processing, offering a high potential for advancing high-performance optically gated photonic devices. Applications include anti-counterfeiting measures, identification, traceability of goods for security, and light-controlled molecular and fluorescent switches.

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ЗАПИС ОПТИЧНОЇ ІНФОРМАЦІЇ НА ОСНОВІ ФОТОКАТАЛІТИЧНОГО ЕФЕКТУ НА НАНОЧАСТИНКАХ ДІОКСИДУ ТИТАНУ ТА ПОЛІМЕРНОМУ НАНОКОМПОЗИТІ, ЛЕГОВАНОМУ ЛЮМІНЕСЦЕНТНИМ БАРВНИКОМ

Анотація. *Діоксид титану є найпоширенішою напівпровідниковою речовиною, яку використовують як фотокаталітичний матеріал у самоочисних поверхнях, системах очищення повітря та води, стерилізації, виділення водню та фотоелектрохімічному перетворенні. У цій роботі ми вперше пропонуємо запис оптичної інформації на основі фотокаталітичного ефекту на наночастинках діоксиду титану та люмінесцентному полімерному нанокомпозиті, легованому барвником. Оптична інформація записувалась у вигляді голографічних ґраток. Для запису оптичної інформації на полімерному нанокомпозиті було застосовано голографічні та неголографічні методи. З цією метою було використано зелений лазерний промінь і галогенно-вольфрамову лампу зі смуговим фільтром. У результаті було отримано високощільну оптичну інформацію з оптичною щільністю 500 мм$^{-1}$. Дані, записані на полімерному нанокомпозиті, є стабільними та довговічними.*

Ключові слова: *діоксид титану, полімерний нанокомпозит, люмінесцентний барвник, фотокаталіз, оптична інформація.*