

Research Article

Solvothermal preparation of $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite for catalytic enhancement

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ABSTRACT

Photocatalysis and sonocatalysis are considered promising techniques employed for the degradation of organic pollutants through solar light, visible or UV light irradiation. In this study, $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite was successfully fabricated using solvothermal route and studied for its ability to degrade Rhodamine B (RhB), when exposed to UV light. The as-prepared nanocomposite was characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), and photoluminescence (PL) spectroscopy. The solvothermal method did not modify the crystalline structure of the TiO_2 and $\text{g-C}_3\text{N}_4$ but enhanced the surface area by interlayer delamination of $\text{g-C}_3\text{N}_4$. The nanocomposite exhibited commendable photocatalytic performances with 99% degradation efficiency of RhB attained in 10 min. The enhanced photocatalytic activities were attributable to UV light-harvesting characteristics and the formation of an S-scheme heterojunction system between two catalysts which promotes interfacial charge separation efficiency and longer charge carrier lifespan. Furthermore, $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite showed high sonocatalytic activity for the degradation of RhB. $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite exhibited the best degradation efficiency of 99.5 % against RhB within 8 min under US light irradiation. Sonocatalytic degradation of organic dye using prepared nanocomposite could be described by the mechanisms of hot spots and sonoluminescence. Based on results, the photocatalytic degradation of RhB via $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite needs more time. The reusability of $\text{TiO}_2/\text{g-C}_3\text{N}_4$ was also examined in order to assess the composite sustainability.

KEYWORDS

Nanocomposite, Photocatalysis, Sonocatalysis, Reusability.

1. Introduction

With the fast acceleration of industrialization, water pollution has emerged as an increasingly pressing global challenge [1]. A significant volume of complex and toxic contaminants, particularly textile dyes and antibiotics, are discharged into aquatic environments [2]. These pollutants are not only persistent and carcinogenic but also have the potential to accelerate the formation and dissemination of antibiotic resistance genes (ARGs), posing severe threats to human health and the stability of ecosystems [3]. Also, heavy metal contamination in water sources can lead to vulnerable diseases, including poisoning and damage to liver and kidney functions [4]. Consequently, the development of efficient, clean, and secondary pollution-free water treatment processes has become a critical and urgent issue that demands immediate attention. Conventional water treatment processes, such as biodegradation, physical adsorption, membrane separation, and chemical oxidation have demonstrated certain efficacy to some extent [5-7]. However, these techniques are often plagued via intricate procedures, high energy consumption, and the potential for secondary pollution. To address these limitations, advanced oxidation processes (AOPs) have garnered increasing attention. Prominent AOPs include ozonation, Fenton reactions, photocatalysis, and electrochemical oxidation [8]. Among all the methods, photodegradation of pollutants has attracted a lot of attention due to its notable advantages including ease of operation, simple instrumentation, nonselective oxidation, cost effectiveness, renewability, and eco-friendliness [9]. In the photocatalytic technique, the energy of light excites electrons from the valence band (VB) to the conduction band (CB), creating electron-

hole pairs. These photogenerated electrons and holes help in the removal of dyes and industrial pollutants. ZnO, CdS, CuO, V₂O₅, TiO₂, and many other semiconductor photocatalysts are now being used to break down dye molecules [10]. Among various semiconductor photocatalysts, TiO₂ has been proven an effective catalyst due to its positive features like its exceptional oxidizing power, photocatalytic performance, cost-effectiveness, low toxicity, and photochemical resistance. The large band gap of TiO₂ (3.20 eV) limits its efficiency, but it is a very effective photocatalyst under UV light radiation [11]. To overcome these challenges, extensive research is needed to improve photocatalytic efficiency by tuning the optical band gap and other properties.

Recently, the coupling of TiO₂ with g-C₃N₄ has gained considerable attention owing to their well-matched band position levels. Graphitic carbon nitride (g-C₃N₄) is known as one of the semiconductor compounds which has received more attention in photocatalysis because of its medium energy bandgap (2.7 eV), nontoxicity, and low generation cost [12]. Its application in photocatalysis is hindered via its low energy band gap, fast recombination of electron-hole pairs, and small surface area. Therefore, the modification of g-C₃N₄ is as significant as those for TiO₂. Most researchers have proved that the coupling of g-C₃N₄ with TiO₂ represents an intriguing solution to enhance the photocatalytic activities [13].

In the last decade, numerous methods have been broadly used to synthesize nanomaterials. Among the various synthetic methods for the nanomaterials, chemical approaches, such as precipitation, sol-gel, microemulsion, electrodeposition, and solvothermal methods, have received much attention because of their simplicity, low cost, ability to make new

materials from the precursors, ability of functionalization, etc.[14, 15]. Solvothermal method is one of the most powerful and widely used methods to synthesize nanomaterials because of the advantages such as high reproducibility, simple procedure, and the easy scale-up, in which precursor solutions are treated in a closed reaction vessel at a high temperature being higher than the boiling point of the solvent [16]. The main intention behind selecting this method is its simplicity and eco-friendly nature, and because high crystallinity and purity of product can be obtained.

The chemical application of ultrasound, “sonochemistry” is a procedure of cavitation that refers to the rapid growth and implosive collapse of bubbles in a liquid in an unusual reaction environment. In recent years, sonocatalysis, which is a combination of solidcatalyst with ultrasonic has received significant interest. The sonocatalytic performance to degrade organic pollutants can be increased because of a synergistic effect between the ultrasonic irradiation and the solid semiconductor catalyst [17].

The aim of the present work was to prepare $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite by the solvothermal method and investigate its catalytic activity for the degradation of a cationic dye (RhB). To find out which energy source is better in terms of the synergistic effect with $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite for the degradation of RhB, UV-C light and US waves were applied as sources of energy. In addition, the photodegradation and sonodegradation mechanisms of RhB on $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite were discussed. The suggested mechanism of RhB sonodegradation involves the sonochemical oxidation of RhB molecules via $\text{OH}^\bullet/\text{HO}_2^\bullet$ radicals in solution and RhB oxidation at the surface of $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite in the presence of O_2 activated

by the cavitation event. These results are being compared herein with those of the photocatalytic RhB degradation results. To the best of our knowledge, there is no report on the comparison of the effect of energy source for the degradation of RhB using $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite.

2. Experimental

2.1. Chemicals

In this work, TiO_2 nanoparticles were prepared using several reagents, including titanium n butoxide (TBOT, $\text{Ti}(\text{OC}_4\text{H}_9)_4$), glacial acetic acid (AcOH). Melamine, sulfuric acid and Rhodamine B (RhB) were purchased from Merck, Germany. All chemicals were of AR grade and used as received. Deionized water was used for all aqueous reactions.

2.1.1. Preparation of TiO_2 nanoparticles by sol-gel low-temperature method

In order to synthesize TiO_2 nanoparticles by sol-gel low-temperature method, TBOT was slowly dissolved in AcOH. After stirring for 5 min at 0 °C, distilled water was added drop by drop to the solution under magnetic stirring. The molar ratio of TBOT/AcOH/ H_2O was 1:1:200. The homogeneous solution was yielded after continuously stirred for 1 h. Then, the obtained sol-solution was kept in darkness for 12 h for nucleation process. The obtained solution was placed in an oven at 70 °C for gelation procedure. The gel was then dried at 100 °C for 24 h. Finally, the dry gel powder was calcined at 400 °C for 3 h.

2.1.2. Synthesis of $\text{g-C}_3\text{N}_4$ nanosheets

$\text{g-C}_3\text{N}_4$ Nanosheets were prepared by chemical exfoliation method [18]. First, 5 g of melamine powder was heated up to 550°C at a ramping rate of 2 °C/min, kept at 550 °C for 4 h, cooled down

to room temperature, and then the yellow bulky g-C₃N₄ was obtained. Then the asprepared bulky g-C₃N₄ was in sequence ground into powder, mixed with H₂SO₄ (98 wt%) in a beaker, stirred in deionized water, ultrasonicated for exfoliation, and then centrifuged and dried at 80 °C. Finally, the porous-layered structural g-C₃N₄ nanosheets were obtained.

2.1.3. Preparation of TiO₂/gC₃N₄ nanocomposite by solvothermal method

TiO₂ and g-C₃N₄ were coupled via the solvothermal method. The TiO₂- g-C₃N₄ mixture (2:1 ratio, 1 g) was stirred for 3 h in ethanol (70%) in a 50 mL PTFE pressure-tight liner at ~65 °C. Subsequently, the PTFE liner was placed in a stainless steel jacket and heated to 180 °C for 10 h. The resulting catalyst was filtered and dried before usage.

2.2. Characterization of prepared samples

The crystal phase composition and the size of prepared samples were analyzed by X-ray diffraction (XRD) (Siemens/D5000) with Cu K α radiation (0.15478 nm) in the 2 θ scan range of 10°–70°. The mean particle size (D in nm) was estimated by Scherrer's formula [19]:

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (1)$$

where k is a constant equal to 0.89, λ , the wavelength of the X-ray equal to 0.154056 nm, β , the full width at half maximum intensity (FWHM) and θ , the half diffraction angle of the peak. The surface morphology of the prepared nanocomposite was obtained via scanning electron microscope (SEM) (Philips XL-30ESM). The chemical composition of the synthesized nanocomposite was analyzed by an energy dispersive X-ray spectroscopy (EDX) system. The fluorescence spectrophotometer

(FLS920, Edinburgh Instruments) was used to measure the photoluminescence (PL) spectra, and the excited wavelength was 320 nm.

2.3. Studies and analysis

2.3.1. Photocatalytic degradation of RhB

The photocatalytic activities of as-prepared photocatalysts were determined by assessing their ability to degrade rhodamine B (RhB) using a 15W (UV-C) mercury lamp (Philips, Holland) emitted around 254 nm. A photolysis experiment was conducted on an aqueous RhB solution under UV light illumination for 20 min. Adsorption and photocatalytic experiments were performed by dispersing 40 mg of each photocatalyst in 100 mL of a 20 mg/L RhB aqueous solution at room temperature. A dark adsorption experiment was conducted for 25 min to establish the adsorption–desorption equilibrium between RhB and the photocatalysts. The zero time reading was yielded from blank solution stored in the dark. Throughout the experiments, vigorous magnetic stirring was applied. At specific time intervals during each experiment, 2 mL aliquots were withdrawn from the solution and subjected to centrifugation to separate the photocatalyst. The absorbance of RhB was measured at its maximum absorption wavelength ($\lambda = 554$ nm) using UV-vis Perkin-Elmer 550 SE spectrophotometer.

2.3.2. Sonocatalytic degradation of RhB

The sonocatalytic performance of the as-prepared TiO₂/gC₃N₄ nanocomposite was evaluated via the degradation of RhB in aqueous solutions at room temperature using an ultrasonic bath (Sonic 6MX, England) under an output power of 300 W. In a typical experiment, 100 mL of an aqueous solution containing RhB dye (20 mg L⁻¹), and 40 mg of the TiO₂/gC₃N₄

nanocomposite powder were added into a 100 mL Pyrex cylindrical vessel. The mixture was vigorously stirred for 30 min to establish the adsorption–desorption equilibrium. Then, it was exposed to ultrasonic irradiation. The sonocatalytic system was maintained at room temperature by a flow of cooling water during the reaction. At regular time intervals, 2 mL of samples of the reaction solution were separated, and the catalyst powder was immediately separated from the suspension with an external magnet, washed and dried at 80 °C for the next runs. Then, the residual RhB concentration in solutions was determined using a UV-vis spectrophotometer at wavelength of 554 nm. However, for the batch adsorptive removal of RhB by TiO₂ /gC₃N₄ nanocomposite without light radiation, the setup was placed inside a fully covered box in order to inhibit any exposure toward light radiation. The percentage degradation of RhB was estimated using Eq. (2):

$$\text{Degradation \%} = \frac{A_0 - A}{A_0} \times 100 \quad (2)$$

where A_0 and A refer to the initial absorbance and absorbance at a specific irradiation time t , respectively [20].

A pseudo-first-order kinetic model was utilized to determine the apparent degradation rate constant (k_{app}) of RhB over TiO₂ /gC₃N₄ [20]:

$$\ln \frac{A_0}{A} = k_{app} t \quad (3)$$

where k_{app} (min⁻¹) is the rate constant and t (min) is the specific time interval of irradiation.

Additionally, 4 cycles of repetition were carried out to evaluate the stability and reusability of the TiO₂ /gC₃N₄ nanocomposite.

3. Results and discussions

3.1. Characterization of prepared nanocomposite

3.1.1. X-ray diffraction

X-Ray diffraction (XRD) is a reliable and widespread identification method especially for crystalline compounds [21]. To investigate the changes in the crystal structure of prepared samples, XRD analysis was carried out. The clear, sharp peaks of the diffraction pattern and low background noise imply that the obtained product is of high purity. From Figure 1 (a), the graphitic carbon nitride has major intensive peaks at 13.05 and 27.5°, corresponding to the (100) and (002) planes (JCPDS card no. 87–1526) [22]. These are related to the planar, which repeats the cell structure and sandwich stack reflection of gC₃N₄. As shown in Fig. 1 (b), pure anatase phase can be seen for prepared TiO₂ nanoparticles. The diffraction peaks of pristine TiO₂ nanoparticles are predominantly in the anatase phase (ICSD File No. 076173) and located at 2θ positions of around 25.3 (101), 37.8 (004), 48.1 (200), 53.9 (105), 55.1 (211), 62.8 (204), and 68.8 (116). The average crystallite size of samples may be estimated via the Scherrer's formula based on the FWHM intensity of anatase (101) phase. The average size of TiO₂ estimated from the XRD pattern based on the Scherrer's equation was about 12 nm. Fig. 1(c) demonstrates XRD pattern of TiO₂/gC₃N₄ nanocomposite. The obvious peak at 27.5° is in line with the pristine g-C₃N₄ sample, indicating the successful incorporation of g-C₃N₄ with TiO₂. In the TiO₂/gC₃N₄ nanocomposite, only (002) peak at $2\theta = 27.5^\circ$ appeared that corresponds to the characteristic peak of g-C₃N₄ for the inter-planar stacking of graphite-like materials. The characteristic (001) peak for the plane of the

tri-s-triazine unit of g-C₃N₄ at around $2\theta = 13.2^\circ$ was not determined due to the low crystallinity of the g-C₃N₄ component [20]. The composite preparation approach did not significantly affect the TiO₂ components, as their peak positions and shape did not change. However, the lower peak intensity could be due to strong interactions between the components, thus destroying the interlayer stacking of g-C₃N₄ and reducing its crystallinity. By the Scherrer equation for spherical particles, we estimated the crystallite size of TiO₂/g-C₃N₄. The average size of crystallites for TiO₂/gC₃N₄ determined from the XRD pattern based on the Scherrer's equation was about 12.8 nm. The TiO₂ crystallite size remains the same or increases only to a limited degree during the composite preparation. On the other hand, the crystallite size for the gC₃N₄ component increases, as would be expected with the improvement of the contact area, whereas g-C₃N₄ with its relative thermal stability would act as a binder between the components.

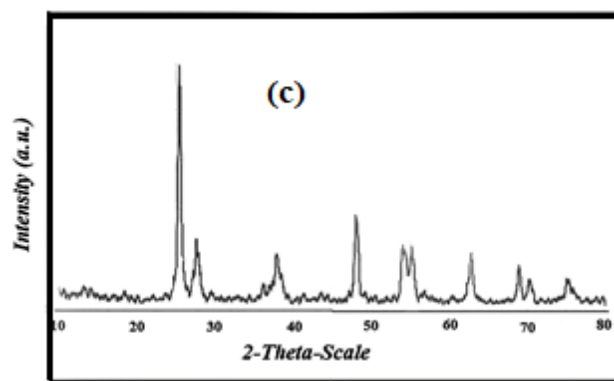
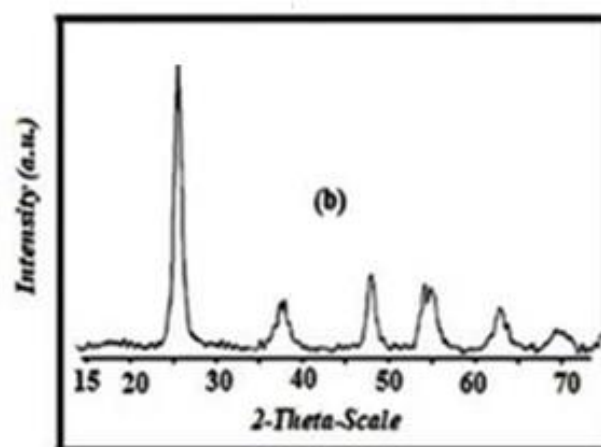
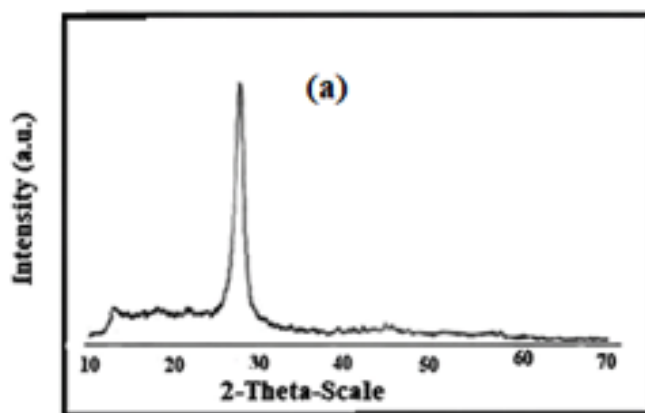


Fig. 1. XRD patterns for the a) g-C₃N₄, b) TiO₂, and c) TiO₂/g-C₃N₄.

3.1.2. Scanning electron microscopy (SEM) analysis

To study the morphological structure of the prepared nanocomposite, SEM analysis was carried out. In the SEM image of TiO₂/g-C₃N₄ nanocomposite (Figure 2) reflects the dispersion of titanium dioxide nanoparticles onto the surface of g-C₃N₄, forming a heterogenous surface with enhanced surface area and intimate interfacial contact, which should increase the transfer of photogenerated electrons and result in higher photocatalytic and electrochemical performance [23].

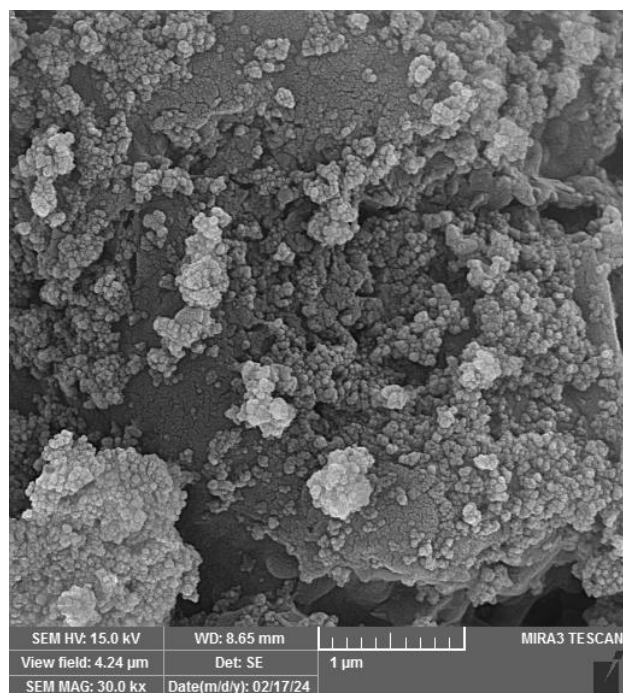


Fig. 2. SEM image of $\text{TiO}_2/\text{g-C}_3\text{N}_4$.

3.1.3. Elemental analysis with EDX spectroscopy

The EDX spectroscopy is used to detect the elemental composition of the prepared samples. C, O, N and Ti peaks can be clearly observed from Fig. 3. EDX analysis showed no significant levels of impurities, which could have originated from the process.

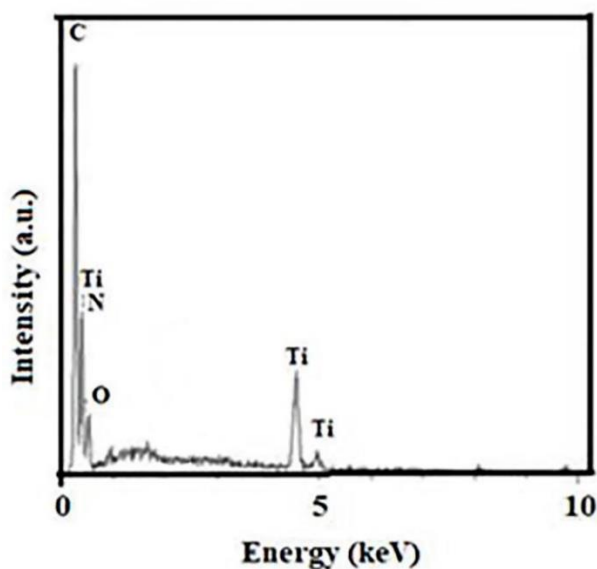


Fig. 3. EDX pattern of $\text{TiO}_2/\text{g-C}_3\text{N}_4$

3.1.4. PL Measurements of $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite

Photoluminescence (PL) analysis was used to investigate the behavior of photoinduced electron-hole pairs in the prepared samples [24]. The intensity of the PL spectra depends on the recombination rate of photogenerated electron-hole pairs. It is commonly believed that the stronger the luminous intensity, the higher recombination rate of charge carriers. Figure 4 demonstrates the PL spectra of $\text{g-C}_3\text{N}_4$, and $\text{TiO}_2/\text{g-C}_3\text{N}_4$ samples obtained at an excitation wavelength of 360 nm. Both $\text{g-C}_3\text{N}_4$ and $\text{TiO}_2/\text{g-C}_3\text{N}_4$ displayed main emission peaks at approximately 460 nm, as shown in Figure 4. A higher PL intensity was observed for $\text{g-C}_3\text{N}_4$ compared to $\text{TiO}_2/\text{g-C}_3\text{N}_4$, implying faster recombination of photoinduced electron-hole pairs in $\text{g-C}_3\text{N}_4$. The decreased PL emission peak of the $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite compared to $\text{g-C}_3\text{N}_4$ can be related to a lower rate of recombination of photogenerated electron-hole pairs, which may be due to the formation of a heterostructure between $\text{g-C}_3\text{N}_4$ and TiO_2 [25].

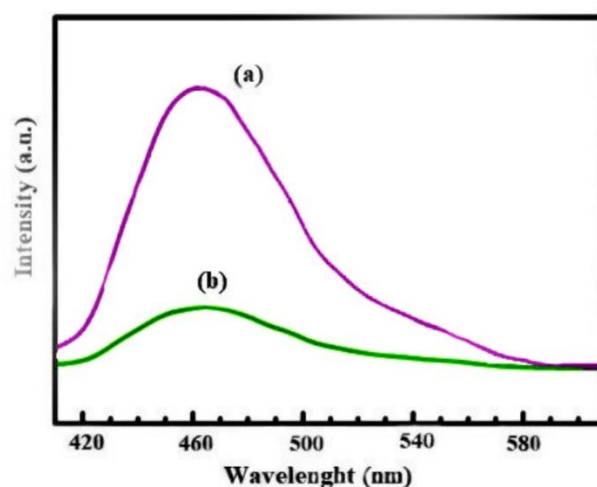
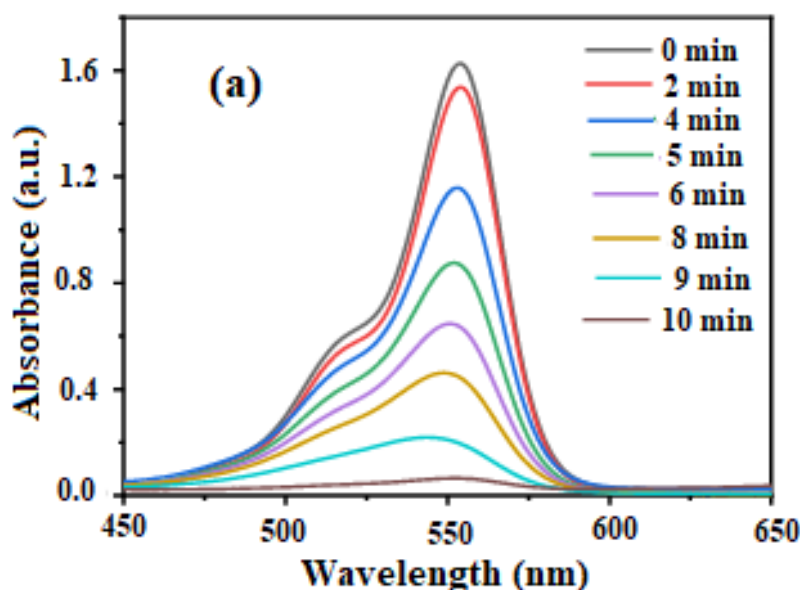


Fig. 4. PL spectra of a) $\text{g-C}_3\text{N}_4$, and b) $\text{TiO}_2/\text{g-C}_3\text{N}_4$.

3.2. Photocatalytic activity of prepared samples

The photocatalytic performance of synthesized samples was investigated via monitoring the main absorption peak of RhB at 554 nm under identical conditions (i.e. 100mL of 20mg L^{-1} of RhB concentration, catalyst dose of 40 mg, pH 6,7, at room temperature, UV-C 254nm, , intensity 2.61mW/cm^2). Fig. 5 (a) demonstrates the UV-vis spectral changes of RhB aqueous solution in the procedure of photodegradation by $\text{TiO}_2/\text{g-C}_3\text{N}_4$. From this figure, it is observed that, the absorbance at 554 nm of RhB decreases gradually with time elapsed: which implies the reduction of RhB from blue to colorless. The absorption peak completely disappeared in 10 min which confirms the complete degradation of RhB. The comparison of the photodegradation rates is given in Fig. 5 (b) together with the adsorption and photolysis study tests. The photolysis study indicates that RhB degradation is photocatalyst-assisted procedure since only a

slight removal of RhB was observed in the absence of a photocatalyst. The adsorption study tests were carried out for $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite and the results show that the degradation of RhB is a light-supported system, since only 21% (RhB) removal efficiency was attained after 10 min. The degradation efficiency of RhB over $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite were obtained to be and 99% (10 min), estimated by equation (2). In the comparison, $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite show more degradation efficiency of RhB as compared to pure TiO_2 and pristine $\text{g-C}_3\text{N}_4$. The improved photocatalytic activity of the $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite is related to the enhancement in photogenerated charge separation because of the development of the S-scheme heterojunction structure.



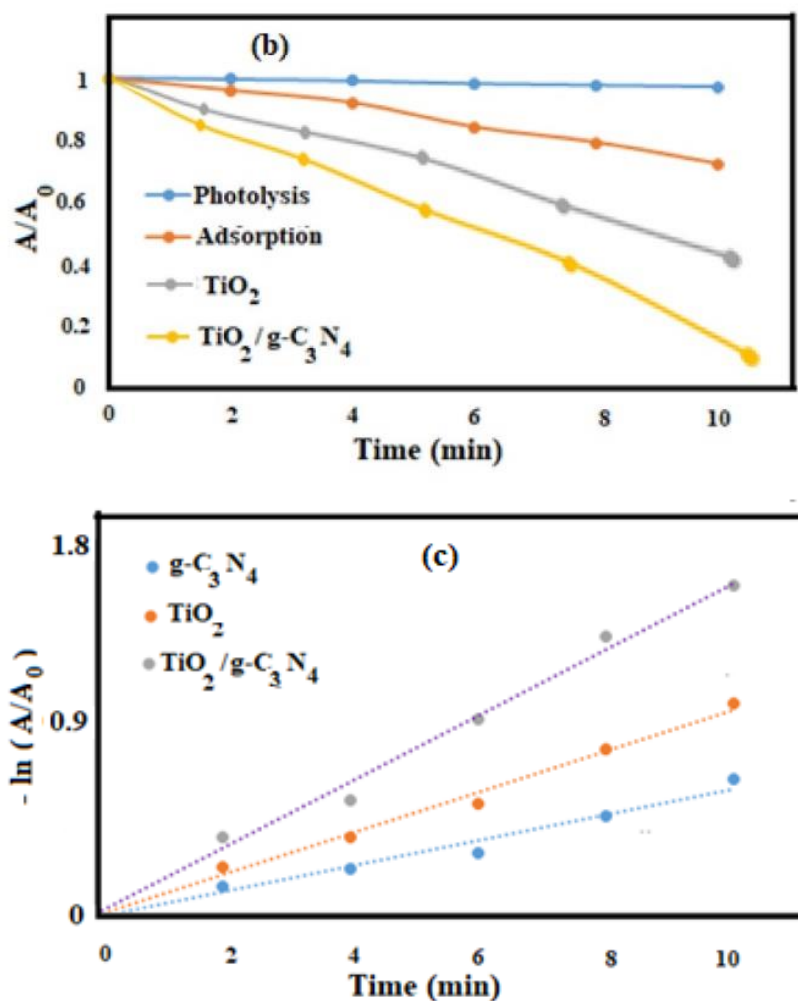


Fig. 5. a) UV-vis absorbance spectra of RhB, b) The photocatalytic degradation rate of RhB, and c) The pseudo-first-order kinetic plots of RhB over synthesized photocatalyst.

Fig. 5 (c) illustrates the linear pseudo-first-order kinetic reaction of RhB. The photocatalytic degradation reaction rate constants for RhB are 0.44, 0.23, and 0.17 min^{-1} for $\text{TiO}_2/\text{g-C}_3\text{N}_4$, TiO_2 ,

and $\text{g-C}_3\text{N}_4$, respectively. Table 1 lists the apparent rate constants of the developed photocatalysts as well as a summary of photodegradation efficiency.

Table 1. Summary of photocatalytic degradation activities of RhB: degradation efficiency, and apparent rate constant (k_{app}) over synthesized photocatalysts.

Photocatalysts	Degradation efficiency (%)	K_{app} (min ⁻¹)
$\text{TiO}_2/\text{g-C}_3\text{N}_4$	99	0.44
TiO_2	60	0.23
$\text{g-C}_3\text{N}_4$	41	0.17

3.2.1. Mechanism of photocatalytic degradation

The increased photocatalytic activity can be explained to the production of heterojunction between TiO_2 and $\text{g-C}_3\text{N}_4$. Based on the

experimental results, possible mechanism of the RhB photodegradation over $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite under UV radiation was proposed. To show matching of energy levels of

the TiO₂ and g-C₃N₄, their valence band (VB) and conduction band (CB) edge potentials were estimated via the Mulliken electronegativity theory by the following equations [26]:

$$E_{VB} = X - E^e + 0.5 E_g \quad (4)$$

$$E_{CB} = E_{VB} - E_g \quad (5)$$

where X and E_g denotes the absolute electronegativity and band gap, respectively. E^e is the energy of free electrons on the hydrogen scale (4.5 eV). Herein, the CB and VB edge potentials of g-C₃N₄ were calculated as -1.08 and +1.55 eV, respectively, while those for TiO₂ were calculated as -0.3 and 2.92 eV, respectively (represented in Fig. 6). Due to their narrow band gaps, under the UV light radiation, TiO₂ and g-C₃N₄ are excited to generate some electron-hole pairs. On the basis of the above results, an effective transfer channel can be constructed for the photogenerated charge carriers in TiO₂/g-C₃N₄ nanocomposite as follows:

Since the CB of the g-C₃N₄ (-1.08 eV) is lower than CB of TiO₂ (-0.3 eV), so the excited-state electrons in the CB of g-C₃N₄ can be easily transfer into CB of TiO₂ [27]. Furthermore, due to VB position of TiO₂ (2.92 eV) is more than of g-C₃N₄ (+1.55 eV), the formed photogenerated holes (h^+) at the VB of TiO₂ will inject to that of g-C₃N₄. Resultantly, the recombination of photoinduced charge carriers is inhibited. The photoelectrons in the conduction band of TiO₂ undergo a reaction with the molecular O₂ to yield O₂^{•-} which oxidizes the pollutant. The O₂^{•-} undergoes a reaction with water and produces HO[•] radicals. At the same time, the photoinduced holes (h^+) in the valence band of g-C₃N₄ undergo a reaction with the H₂O yield HO[•] radicals. The redox potential of HO[•]/H₂O (2.68 eV vs. NHE) is less positive than the VB potential of TiO₂ (2.92 eV) so the VB holes of

TiO₂ can oxidize the water molecules to form OH[•] radicals. The HO[•] and O₂^{•-} radicals being strong oxidizing agents degrade the pollutant molecules into nontoxic end products such as H₂O and CO₂ [28]. Based on the above findings, an S-scheme heterojunction photocatalytic mechanism can be constructed as depicted in Fig. 6. The photochemical degradation procedure can be explained in Eq. (6- 11) [29]:

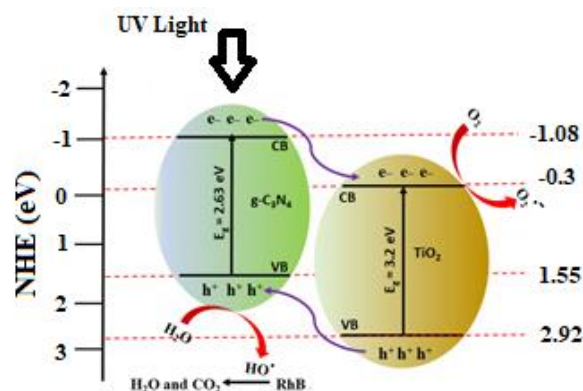
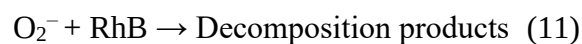
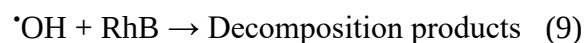


Fig. 6. Mechanism of catalytic degradation of RhB.

3.3. Effect of scavengers in photocatalytic performance

The process of photocatalytic degradation of most pollutants can be easily performed in the presence of radicals and oxidizing species [30]. For this purpose, by using EDTA, Isopropanol, and Ascorbic acid as scavengers, which are used to trap h^+ , OH[•], $\cdot\text{O}_2^-$ species, respectively, it is possible to predict the photocatalytic degradation mechanism of Rh.B in the presence of the TiO₂/g-C₃N₄ nanocomposite. As the Fig. 7

shows, the lowest amount of photocatalytic degradation occurred in the presence of Ascorbic acid, in other words, $\cdot\text{O}_2^-$ species is the most important effective factor and driver of the photocatalytic degradation mechanism of the

Rh.B dye. The results showed that Ascorbic acid has more significant impact, followed by Isopropanol and finally EDTA. In other words, the oxidant effects on RhB degradation in the order of $\cdot\text{O}_2^- > \cdot\text{OH} > h^+$ [31].

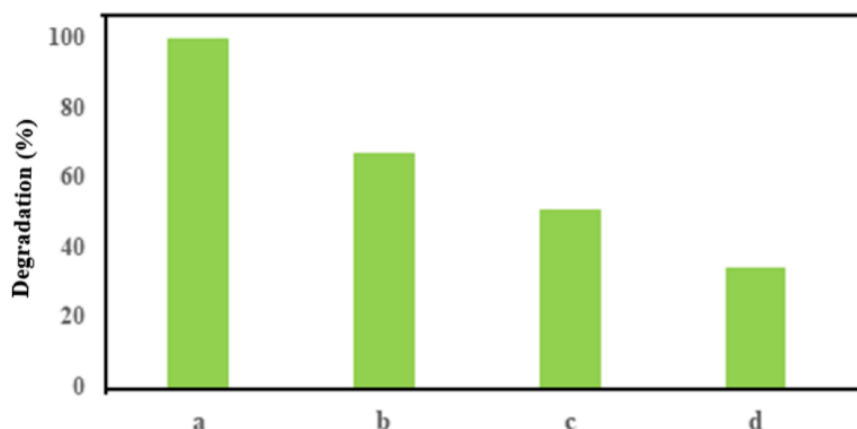


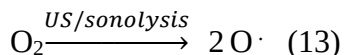
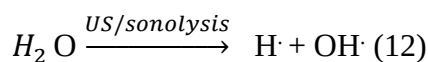
Fig. 7. The effect of radical scavengers on the photocatalytic degradation of RhB by $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite: (a) No quencher, (b) EDTA, (c) Isopropanol, and (d) Ascorbic acid.

3.4. Sonodegradation of RhB via prepared nanomaterials

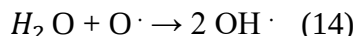
The sonocatalytic activity of $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite was investigated in degradation of RhB dye in aqueous solution under ultrasonic irradiation. The process was investigated by monitoring the characteristic absorption band of RhB at about 554 nm. Initially, a suspension of 40 mg catalyst in 100 mL of aqueous solution RhB (20 mg L^{-1}) at pH 6,7 was exposed to ultrasonic (US) irradiation. RhB undergoes few degradation under direct ultrasonic radiation in the absence of $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite. This may be related to the low rate of hydroxyl radical generation via sonolysis alone. However, the presence of $\text{TiO}_2/\text{g-C}_3\text{N}_4$ exhibited excellent results as a sonocatalyst in the removal of RhB (Figure 8). This phenomenon can be attributed to the increased number of cavitation bubble which occurs on the surface of $\text{TiO}_2/\text{g-C}_3\text{N}_4$ and yielded in water

cleavage and production of excess hydroxyl radicals. Also, the fast degradation of RhB via sonocatalytic process in the presence of $\text{TiO}_2/\text{g-C}_3\text{N}_4$ can be attributed to the sonoluminescence mechanism. Sonoluminescence involves an intense UV-light, which promotes $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite to act as an efficient photocatalyst during ultrasonic radiation [31]. The sonodegradation mechanism of RhB on $\text{TiO}_2/\text{g-C}_3\text{N}_4$ can be expressed as follows:

The chemical influences of ultrasound include the procedure of cavitation which is the nucleation, growth, and collapse of bubbles in a liquid [17]. The collapse of the bubbles induces high-energy phenomena, i.e., high temperature and pressure ($\sim 5000 \text{ K}$ and 500 bars), electrical discharges, and plasma effects. The consequences of these extreme conditions are the direct thermal dissociation (sonolysis) of dissolved oxygen and water molecules into highly reactive radical species:



O₂[·] can react with water molecules to form OH[·]:



OH[·] can easily degrade RhB molecules:



Also, sonochemistry includes the emission of light energy for a small period of time. The irradiation can lead to the photoexcitation of electrons from the valence band to the conduction band, thus leading to the formation of electron-hole pairs in a similar manner as explained above for the photocatalysis [32]. Hydroxyl radicals with high oxidizing activity can more react with RhB and degrade it into small species. The organic dye decomposition mechanism involved:

- Adsorption, the electrostatic attraction between g-C₃N₄ and RhB molecules is a key factor in the adsorption process. The g-C₃N₄, exhibit a high rate of adsorption for cationic dyes like RhB. This high rate is attributed to the electrostatic interaction between the negatively charged g-C₃N₄ and the cationic organic dye molecules.

- Sonocatalysis, the sonocatalytic degradation of RhB by ultrasonic radiation began with excitation of TiO₂/g-C₃N₄ and formation of electron-hole pairs. High oxidation valence-band holes can oxidize dye molecules. Water degradation or reaction of h⁺ with OH[·] can form hydroxyl radicals. Meanwhile, the reaction between conduction-band electrons (e⁻) and proper electron acceptors (such as O₂) yielded oxidative radicals. The produced hydroxyl radicals can easily degraded dye molecules [33].

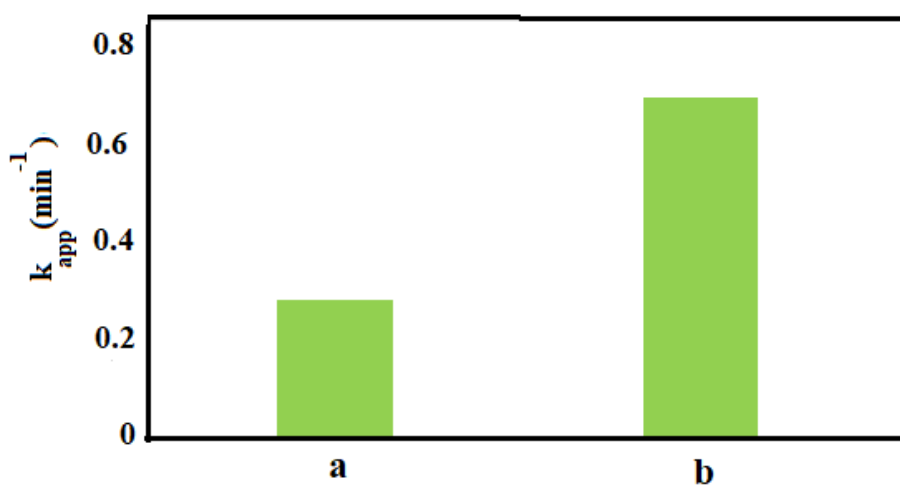


Fig. 8. Sonocatalytic degradation of RhB in the presence of (a) US only, (b) TiO₂/g-C₃N₄.

3.5. Comparison of Efficiency of Photocatalysis and Sonocatalysis of TiO₂/g-C₃N₄ nanocomposite

The degradation rate of RhB by photolysis and sonolysis procedure in the presence of TiO₂/g-C₃N₄ nanocomposite is exhibited in Fig. 9. It can be seen that sonodegradation process is faster

than photodegradation process. Therefore, US irradiation is a better source than UV irradiation for the degradation of organic pollutants in the presence of TiO₂/g-C₃N₄ nanocomposite. TiO₂ nanoparticles have a lower band gap (3.2 eV). So, electron-hole recombination is faster and easier. Therefore, the formation of OH[·] is hard,

which results in lower degradation of RhB in the case of photocatalysis. In the presence of $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite, the sonodegradation rate of pollutant was increased. This is because of synergistic effect of ultrasound and solid catalyst, namely [33], (1) added materials could provide additional nuclei for cavitation bubble formation, (2) US radiation enhances the mass transfer of RhB between the liquid phase and $\text{TiO}_2/\text{g-C}_3\text{N}_4$

nanocomposite surface, (3) US radiation increases the active surface area because of ultrasound disaggregating, and (4) the catalyst can be promoted via ultrasound-induced luminescence which has a wide wavelength. This phenomenon can enhance the production of $\text{OH}^\cdot$ in the reaction mixture. Therefore, sonocatalysis can increase the degradation rate of RhB.

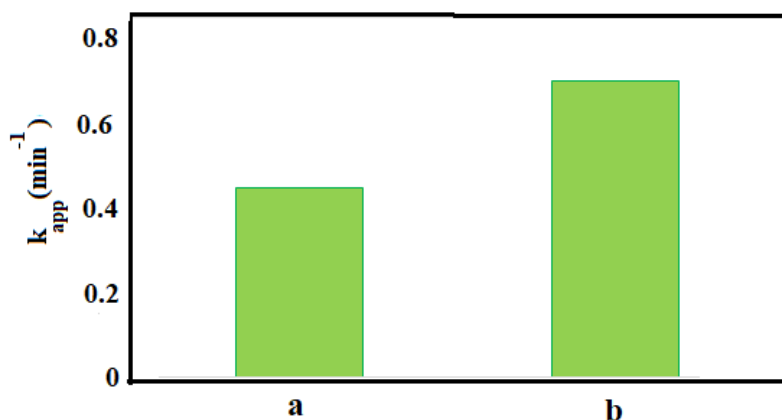


Fig. 9. Degradation rates of RhB under (a) UV-visible radiation and (b) US radiation.

3.6. Comparative study

The literature suggests that the catalytic performance of materials is influenced by various factors, including the catalyst's physical and chemical properties, wavelength, light source, initial pollutant concentration, catalyst dose, and other experimental conditions. The comparison of the current study with several previous literature reports is shown in Table 2. According to the previous report [34], $\text{TiO}_2/\text{g-C}_3\text{N}_4$ was synthesized via wet-chemical method. The as-synthesized $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite was used for photodegradation of a 100mg L^{-1} Basic Blue 3 dye (BB3) solution with a catalyst dose of 50mg. The photodegradation experiments were conducted under a UV-C 254 nm lamp equipped with intensity 2.57mW/cm^2 . After 240 min of irradiation, $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite achieved 99.68 % BB3 removal. Thermodynamic

studies demonstrated that the photodegradation of BB3 dye over $\text{TiO}_2/\text{g-C}_3\text{N}_4$ is a non-spontaneous process. Likewise, $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite was synthesized by hydrothermal method and used for photodegradation of RhB in aqueous media. The catalyst demonstrated a 95.2 % removal efficiency for 10^{-5} M dye within 80 min [35]. Likewise, $\text{TiO}_2/\text{g-C}_3\text{N}_4$ was synthesized using a solvothermal method and subsequently employed for the removal of Bisphenol A and Malathion pesticides. The composite material exhibited removal efficiencies of 71 and 50% for Bisphenol A and Malathion pesticides, respectively. The photocatalytic degradation process was monitored using a pseudo-first order kinetic model (PSO) [36]. Similarly, $\text{TiO}_2/\text{g-C}_3\text{N}_4$ composites were synthesized by calcination method and employed for the degradation of MB and RhB dyes under 300 W

Xenon lamp irradiation. Utilizing 20 mg of catalyst, degradation efficiencies of 90 % for 30 ppm MB and 93.13% for 15 ppm RhB were achieved after 240min of slight exposure. The photocatalytic degradation process followed pseudo-first-order kinetic model [37]. Additionally, TiO₂/g-C₃N₄ composites were synthesized using Pyrolysis method and applied for the degradation of RhB dye. The 97% removal was found for RhB dye of 10ppm concentration under 300 W Xenon lamp radiation for 80min. Additionally, the

composites exhibited excellent stability, maintaining high performance after four cycles of reuse [38]. In this work, the TiO₂/g-C₃N₄ composite showed significantly higher removal percentage of 99 % for RhB dye compared to the pristine g-C₃N₄, and TiO₂ with removal % of 41, and 60 %, respectively. The prepared composite revealed excellent photodegradation and sonodegradation efficiencies in a shorter time. Additionally, the as-synthesized nanocomposite exhibits high catalytic activity even after 5 consecutive recycles.

Table 2. The distinction between the current study and previous studies on the synthesis of TiO₂/g-C₃N₄ photocatalyst for the photodegradation of organic dyes.

Synthetic method	Pollutant	Concentration of Pollutant	Degradation Efficiency	Light source	Irradiation time,	Refs.
wet-chemical method	BB3	100 mg L ⁻¹	99 %	UV	240 min	[34]
Hydrothermal	RhB	10 ⁻⁵ M	95.2 %	Visible light	80 min	[35]
Solvothermal	Bisphenol A	20 mg L ⁻¹	71 %	Visible light	1.0 min	[36]
	Malathion pesticides		50 %			
Calcination method	RhB + MB	30 mg L ⁻¹	90 %	300 W Xenon lamp	240 min	[37]
Pyrolysis	RhB	10 mg L ⁻¹	97 %	300 W Xenon lamp	40 min	[38]
Solvothermal	RhB	20 mg L ⁻¹	100 %	UV US	10 min 7.5 min	This work

3.7. Kinetic study as function of initial dye concentration

The influence of initial dye concentration was investigated in the range of 10– 40 mg L⁻¹. The plot of ln (C₀/C) vs. time for all the tests with various dye concentration is shown in Fig. 10. The values of k_{app} can be obtained by applying a least square regression analysis. The apparent rate constant values for photodegradation of 10, 20, 30, and 40 mg L⁻¹ RhB by TiO₂/g-C₃N₄

nanocomposite are 0.39, 0.21, 0.1, and 0.072 min⁻¹ respectively. furthermore, the k_{app} values for sonodegradation of 10, 20, 30, and 40 mg L⁻¹ RhB are 0.65, 0.41, 0.24, and 0.17 min⁻¹ respectively. It is observed that the value of k_{app} is decreased by increasing initial dye concentration. This behavior can be due to following reasons: (i) an increase in competition between the intermediates and the parent compounds for the reaction with reactive

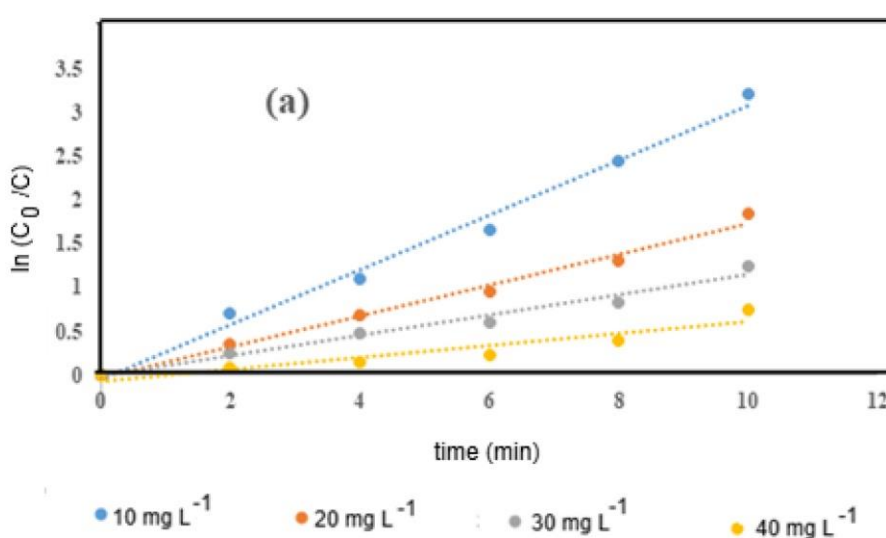
oxidizing radicals, (ii) decreasing the generation rate of reactive species, because of filling the reactive sites by RhB molecules and reducing the reactions between US or UV irradiation and catalyst, and (iii) the degradation of all RhB molecules would be impossible when the production rate of reactive oxidizing species is constant. In contrast, at lower concentrations, a sufficient number of reactive oxidizing species is available in the solution for reaction with RhB molecules that leads to high degradation rate. Classic Langmuir–Hinshelwood (L–H) mechanism can be applied to explain the kinetic of the catalytic degradation of most organic dyes which in terms of degradation kinetics can be described as follows [39]:

$$r = \frac{k_C k_{dye} (C)}{1 + k_{dye} (C_0)} \quad (16)$$

$$\frac{1}{k_{app}} = \frac{1}{k_C k_{dye}} + \frac{C_0}{k_C} \quad (17)$$

In these equations, k_{dye} and k_C are the adsorption equilibrium constant and surface reaction rate constant, respectively. k_{dye} and k_C can be calculated using the resulted data from

photocatalytic and sonodegradation tests with different initial dye concentrations and plotting $1/k_{app}$ vs. C_0 . As can be seen in Figure 11, a straight line fitted the experimental data reasonably well, thus indicating that degradation of RhB by $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite most probably follows Langmuir–Hinshelwood kinetics. From the slope and the intercept of this line, k_C and k_{dye} were computed. Table 7 reports the values of k_C and k_{dye} for both method resulting from plot of $\frac{1}{k_{app}}$ vs. “ C_0 ”. Kahoul and his coworkers reported Langmuir–Hinshelwood equation constants for photodegradation of RhB by TiO_2 nanoparticles prepared via a non-conventional sol-gel method under UV irradiation. The values of k_C was $1.56 \text{ mg L}^{-1} \text{ min}^{-1}$, while k_{dye} was $0.0436 (\text{mg L}^{-1})^{-1}$ [40]. Moreover, Dogdu and his coworkers reported Langmuir–Hinshelwood equation constants for decolorization of RhB dye by sonophotocatalysis method via TiO_2 nanoparticles. They found that the Langmuir–Hinshelwood (L-H) model could clarify the sonophotocatalysis process well, where k_C and k_{dye} were $0.941 \text{ mg L}^{-1} \text{ min}^{-1}$ and $0.129 (\text{mg L}^{-1})^{-1}$, respectively [41].



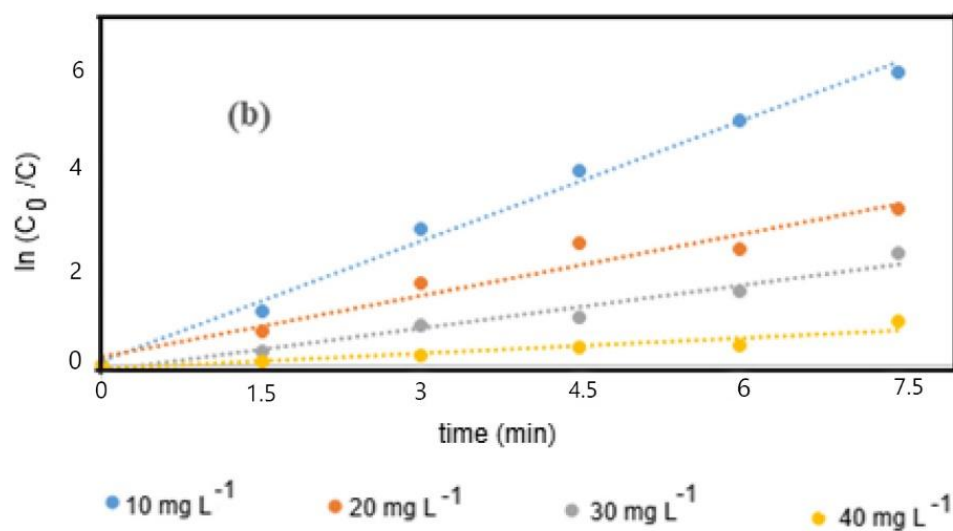


Fig. 10. Determination of the pseudo-first-order kinetic rate constant, k_{app} :
(a) Photodegradation and (b) Sonodegradation methods.

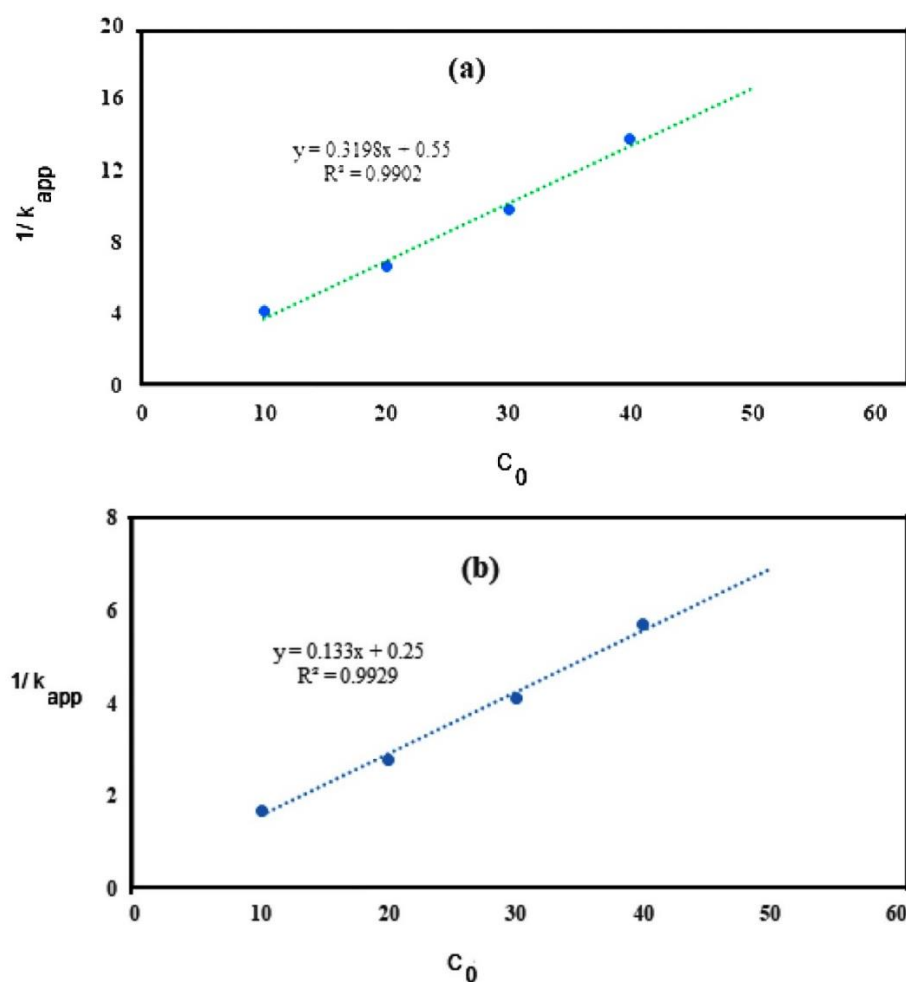


Fig. 11. Determination of the adsorption equilibrium constant, k_{dye} , and the second-order rate constant, k_C , for the Langmuir-Hinshelwood kinetic model: (a) Photodegradation and (b) Sonodegradation methods.

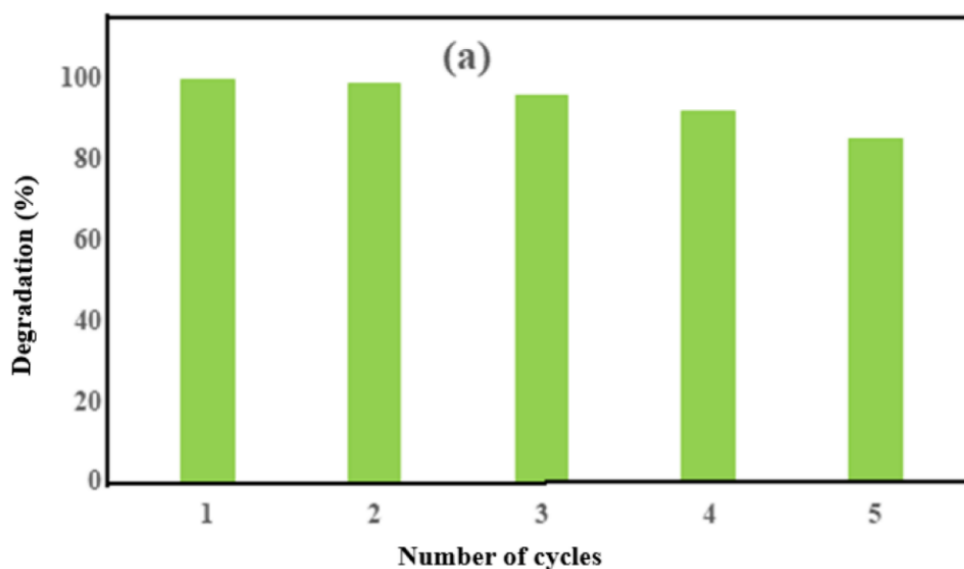
Table 3. k_{dye} and k_{C} values in the degradation of RhB by $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite.

Method	k_{C} ($\text{mg L}^{-1} \text{ min}^{-1}$)	k_{dye} ($(\text{mg L}^{-1})^{-1}$)	R^2
Photodegradation	3.22	0.56	0.990
Sonodegradation	7.5	0.53	0.992

3.8. Reusability and stability of $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite

The prospective applicability of the synthesized catalyst for the large-scale applications can be tested on the basis of the stability and reusability. The reusability of the $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite was repeated for the RhB degradation up to five cycles by taking the 40 mg catalyst in 100 mL dye solution of 20 mg L^{-1} concentration at pH 6,7 and the results are shown in Fig.12a. For the recyclability, $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite was washed every time with de-ionized water and dried at 60°C for 12 h. Figure 9a shows that the $\text{TiO}_2/\text{g-C}_3\text{N}_4$ composite was stable even after five cycles of regeneration and could retain 85.2 % degrading efficiency. The decrease in the degrading efficiency can be due to the loss of

nanocomposite because of washing [42]. Also, it can be ascribed to the adsorbed molecules of RhB or degradation product in the internal structure of the catalyst. XRD analysis was used to examine the stability of the catalysts' crystal structure for $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite before and after the five cycle experiments. The XRD spectra results for $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite before photocatalysis and after five cycles of regeneration were shown in Fig.12b. XRD patterns before and after cycling retain the signature anatase peak with no discernible shift or broadening, indicating preserved crystallinity and absence of rutile formation. These results indicated that $\text{TiO}_2/\text{g-C}_3\text{N}_4$ nanocomposite is a stable catalyst under the applied experimental conditions.



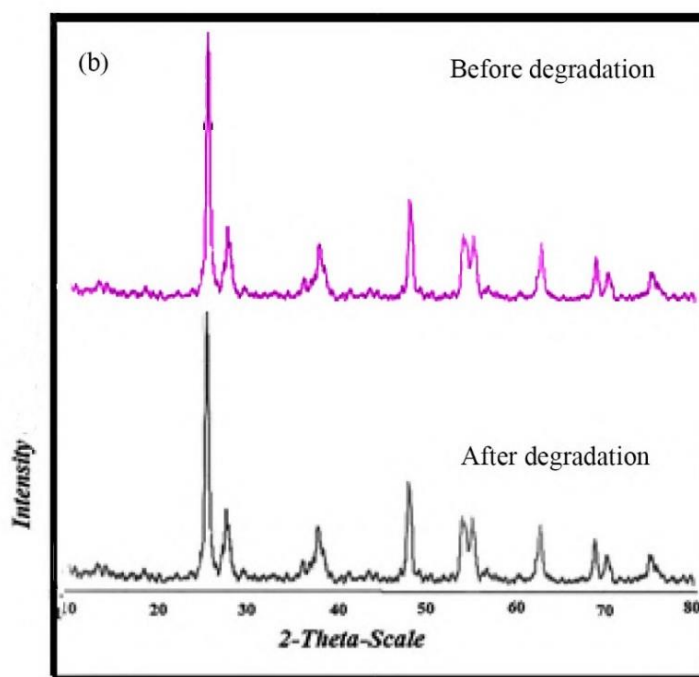


Fig. 12. a) Photocatalytic degradation of RhB by TiO₂/ g-C₃N₄ nanocomposite in five consecutive cycles , and b) XRD spectrum of fresh and reused TiO₂/ g-C₃N₄ nanocomposite.

4. Conclusions

In this work, the degradation of RhB on a formed direct S-scheme TiO₂/g-C₃N₄ heterojunction nanocomposite was presented. The S-Scheme TiO₂/g-C₃N₄ nanocomposite demonstrates higher photocatalytic performance with high degradation efficiency for organic dyes. The enhancement in photocatalytic performance of nanocomposite was accredited to the construction of the S-scheme heterojunction structure between TiO₂ and g-C₃N₄ which promote the enhancement of charge separation. The results showed that the synergistic effect of TiO₂/g-C₃N₄ nanocomposite and sonocatalysis is more effective in degrading of RhB as compared to TiO₂/g-C₃N₄ nanocomposite and photocatalysis. Sonocatalytic degradation of RhB in the presence of TiO₂/g-C₃N₄ nanocomposite could be explained by the mechanisms of hot spots and sonoluminescence. Furthermore, the photocatalytic degradation of RhB by TiO₂/g-C₃N₄ nanocomposite needs more

time. Based on results, the degradation efficiencies of RhB remain above 85.2 % after 5 degradation cycles. This research work simply explored a novel approach for constructing a high efficiency catalyst for water purification.

Acknowledgment

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سنتر سولوترمال نانوکامپوزیت $\text{TiO}_2/\text{g-C}_3\text{N}_4$ به منظور افزایش فعالیت فتوکاتالیزوری

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چکیده

فتوکاتالیز و سونوکاتالیز تکنیک‌هایی هستند که برای تخریب آلاینده‌های آلی از طریق تابش نور خورشید، نور مرئی و UV مورد استفاده قرار می‌گیرند. در این تحقیق نانوکامپوزیت $\text{TiO}_2/\text{g-C}_3\text{N}_4$ توسط روش سولوترمال با موفقیت سنتز شده و قابلیت آن در تخریب رودامین B (RhB) در معرض تابش نور UV مورد مطالعه قرار گرفته است. نانوکامپوزیت سنتز شده با استفاده از پراش پرتو X (XRD)، میکروسکوپ الکترونی روبشی (SEM)، طیف‌سنجی پراش انرژی پرتو ایکس (EDX) و طیف سنجی فتولومینسانس (PL) شناسایی شد. روش سولوترمال ساختار کریستالی TiO_2 و $\text{g-C}_3\text{N}_4$ را تغییر نداد ولی باعث افزایش سطح ویژه $\text{g-C}_3\text{N}_4$ گردید. نانوکامپوزیت سنتز شده با فعالیت فتوکاتالیزوری بالا توانست ۹۹٪ رودامین B را در مدت ۱۰ دقیقه تخریب کند. بهبود فعالیت فتوکاتالیزوری مربوط به مشخصه‌های نور UV و تشکیل سیستم S-scheme بین دو کاتالیزور است که باعث بهبود کارایی جدایش بارها شده و طول عمر حاملین بار را افزایش می‌دهد. همچنین نانوکامپوزیت $\text{TiO}_2/\text{g-C}_3\text{N}_4$ فعالیت سونوکاتالیزوری بالایی را در تخریب رودامین B از خود نشان داد به طوری که باعث تخریب ۹۹.۵٪ رودامین B در مدت ۸ دقیقه تحت تابش US گردید. تخریب سونوکاتالیزوری رودامین B با استفاده از نانوکامپوزیت سنتز شده از طریق مکانیسم سونولومینسانس و نقاط داغ قابل توجه است. طبق نتایج حاصل، تخریب فتوکاتالیزوری رودامین B با استفاده از نانوکامپوزیت $\text{TiO}_2/\text{g-C}_3\text{N}_4$ به زمان بیشتری نیاز دارد. همچنین قابلیت بازیافت نانوکامپوزیت $\text{TiO}_2/\text{g-C}_3\text{N}_4$ به منظور ارزیابی میزان پایداری آن، مورد آزمایش قرار گرفت.

واژه‌های کلیدی

نانوکامپوزیت، فتوکاتالیز، سونوکاتالیز، قابلیت بازیافت.

