

RESEARCH PAPER

Preparation of $\text{TiO}_2/\text{Cu}_{0.4}\text{Fe}_{1.6}\text{Ni}_{0.65}\text{Zn}_{0.35}\text{O}_4$ Nanocomposite for Degradation of Acid Red 88 under Visible Light Irradiation

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ABSTRACT

An excellent nanocomposite comprising TiO_2 and $\text{Cu}_{0.4}\text{Fe}_{1.6}\text{Ni}_{0.65}\text{Zn}_{0.35}\text{O}_4$ (CFNZO) nanoparticles was prepared and demonstrated to be an efficient visible light photocatalyst. To verify photocatalytic potential of the prepared nanocomposite, optical characterizations were carried out using diffuse reflectance (DRS) and photoluminescence (PL) spectroscopy. The nanocomposite containing 2 wt% of CFNZO nanoparticles ($\text{TiO}_2/2\text{-CFNZO}$) showed a band gap value of 2.76 eV. Acid red 88 (AR88) solution was degraded using the prepared nanocomposite under the visible light irradiation for 120 min. The photocatalytic activity of the nanocomposite was carefully examined under various conditions, such as varying content of CFNZO nanoparticles, different concentrations of photocatalyst, AR88 solution pH, and H_2O_2 concentrations. Accordingly, the highest photocatalytic degradation level was achieved in acidic AR88 solution (pH = 3), using 0.04 g of $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite and 2 mL of 0.02 M H_2O_2 oxidant. Additionally, the degradation reactions performed using various radical scavenger agents confirmed that the reaction proceeded via hydroxyl radicals and photo-generated holes. Furthermore, the prepared nanocomposite exhibited the superior stability and reusability, successfully performing the AR88 degradation reaction over 7 consecutive cycles.

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INTRODUCTION

With the escalating issue of water pollution, we are facing an environmental catastrophe [1]. Not only human, but other beings are affected by pollution of oceans and seas [2]. Human activities are a leading cause for this crisis, and annually sheer volume of toxic compounds including industrial, agricultural, and municipal wastewater

are released into environment [3, 4]. As a consequence, numerous life-threatening diseases have emerged, and the sustainability of our food supply is under threat due to water pollution. Therefore, governments and health agencies around the world are spending billion dollars to mitigate these problems [5, 6].

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Fortunately, we have a potential complement to clean water resources, employing photocatalysis method [7, 8]. According to its methodology, photocatalysis is defined by light-activated catalyst, which produces highly reactive oxidative species [9, 10]. These species, including hydroxyl ($\text{OH}^\bullet$) and superoxide ($\text{O}_2^{\bullet-}$) radicals, are sufficiently strong to degrade pollutants. Titanium dioxide (TiO_2) and zinc oxide (ZnO) are two major photocatalysts, which have been widely used. However, low quantum efficiency of TiO_2 and ZnO caused by limited lifetime of photo-generated electrons and holes is a main obstacle to harness photocatalytic method for degradation of pollutants [11-13].

In order to effectively utilize photocatalysis for water purification, it is important to have a superior photocatalyst. Recently, many efforts have been devoted to make an efficient photocatalyst, operating under visible light irradiation [14, 15]. Mixed metal oxides have occupied a prominent position in this regard [16]. Owing to the facilitated electron transitions across the energy levels on the different components, the quantum yield of photocatalyst increases as high as possible [17]. These electronic transitions significantly decrease recombination of the charge carriers—photo-generated electrons and holes—thereby increase their lifetime for participating in the pollutant degradation process [18, 19].

For example, Zhang et al. reported the photocatalytic activity of $\text{CuBi}_2\text{O}_4/\text{CdMoO}_4$ for degradation of methylene blue [20]. Peng et al. synthesized $\text{MgAlO}/\text{MgCr}_2\text{O}_4$ as a mixed metal oxide photocatalyst to oxidize cyclohexane [21]. A ternary mixed metal oxide nanocomposite comprising of $\text{Ag}_2\text{O}/\text{NiO}/\text{ZnO}$ was synthesized for removal of organic pollutant by Bhatia et al. [22].

In this study, $\text{TiO}_2/\text{Cu}_{0.4}\text{Fe}_{1.6}\text{Ni}_{0.65}\text{Zn}_{0.35}\text{O}_4$ nanocomposite ($\text{TiO}_2/\text{CFNZO}$) was prepared via two-step synthetic route. First, a co-precipitation reaction was used to prepare $\text{Cu}_{0.4}\text{Fe}_{1.6}\text{Ni}_{0.65}\text{Zn}_{0.35}\text{O}_4$ nanoparticles (CFNZO) in the presence of polyethylene glycol (PEG) as capping agent. Then, the prepared CFNZO nanoparticles were added to an as-prepared TiO_2 sol solution and the $\text{TiO}_2/\text{CFNZO}$ nanocomposite was prepared by sol-gel reaction. The nanocomposite is expected to serve as the photocatalyst for removal of aqueous solution of acid red 88 (AR88) under visible light source. Therefore, the photocatalytic degradation of AR88 was investigated using by added different amounts of $\text{TiO}_2/\text{CFNZO}$ nanocomposite under the

visible light irradiation. Moreover, the degradation efficiency was studied under other experimental conditions, including varied pH of AR88, different H_2O_2 concentration, and different radical scavenger agents.

MATERIALS AND METHODS

Preparation of $\text{Cu}_{0.4}\text{Fe}_{1.6}\text{Ni}_{0.65}\text{Zn}_{0.35}\text{O}_4$ nanoparticles (CFNZO)

The CFNZO nanoparticles was synthesized using the co-precipitation method. To this end, 1.0 g of polyethylene glycol (PEG) was added to the 100 mL of deionized water, and the metal precursors were added, including $\text{Zn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ (0.35 mmol), $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ (0.4 mmol), $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (1.6 mmol), and $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.65 mmol). The obtained solution was stirred for 30 min. Then, the pH of solution was adjusted to around 9 using addition of NaOH (0.1 M) and stirring was performed for another 30 min. The precipitated product was then collected by centrifugation at 6000 rpm for 10 min and washed several times using 20% (V/V) ethanol solution. Finally, the obtained solid was dried overnight in an oven at 100 °C and then calcined at 600 °C for 7 h.

Preparation of $\text{TiO}_2/\text{Cu}_{0.4}\text{Fe}_{1.6}\text{Ni}_{0.65}\text{Zn}_{0.35}\text{O}_4$ nanocomposite ($\text{TiO}_2/\text{CFNZO}$)

The $\text{TiO}_2/\text{CFNZO}$ nanocomposite was prepared via sol-gel route, as follows: First, 1 mmol of tetrabutyl orthotitanate (TBOT) was added into 50 mL of absolute ethanol, and then appropriate amount of the as-prepared CFNZO nanoparticles (2, 4, and 6 wt%) was added and the mixture was stirred for 2 h. The mixture was dried at 90 °C for 24 h and then calcined at 600 °C for 7 h. The nanocomposites comprising 2wt%, 4wt%, and 6wt% of the CFNZO nanoparticles were signified as $\text{TiO}_2/2\text{-CFNZO}$, $\text{TiO}_2/4\text{-CFNZO}$, and $\text{TiO}_2/6\text{-CFNZO}$, respectively.

Characterization

Surface morphology and elemental composition of the prepared nanocomposite were analyzed via scanning electron microscope (SEM, TESCAN Mira3). Phase structure of nanocomposite was investigated using X-ray diffraction analysis (XRD, Philips Pro MPD). Light absorption properties of the prepared nanocomposite was studied using diffuse reflectance spectroscopy (DRS) by JASCO UV/Vis spectrophotometer. Also, photoluminescence (PL) spectroscopy by PL CARY ECLIPSE to further

investigate the optical properties.

Photocatalytic studies

The photocatalytic activity of the prepared $\text{TiO}_2/\text{CFNZO}$ nanocomposite was tested using degradation of acid red 88 (AR88) solution (50 ppm) under LED light (50 W, $\lambda > 500$ nm) irradiation. The photocatalytic tests were initially

performed in the darkness under stirring for 30 min. It is important to achieve adsorption/desorption equilibrium between nanocomposite particles and AR88 molecules. Then, illumination was carried out from a distance of 25 cm for 120 min, and every 30 min the degradation of AR88 was investigated using VU/Vis spectroscopy at maximum wavelength (506 nm) of AR88 solution.

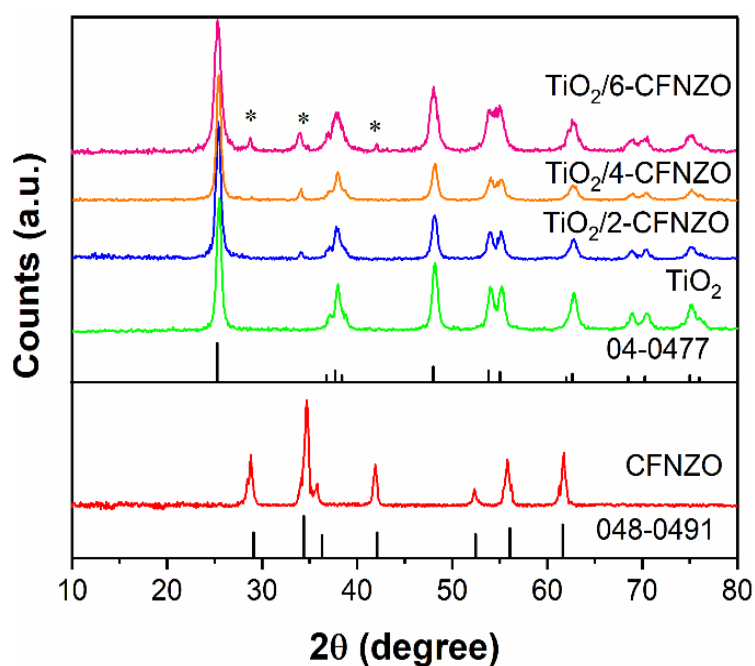


Fig. 1. XRD patterns for pure CFNZO, pure TiO_2 , and different prepared $\text{TiO}_2/\text{CFNZO}$ nanocomposites.

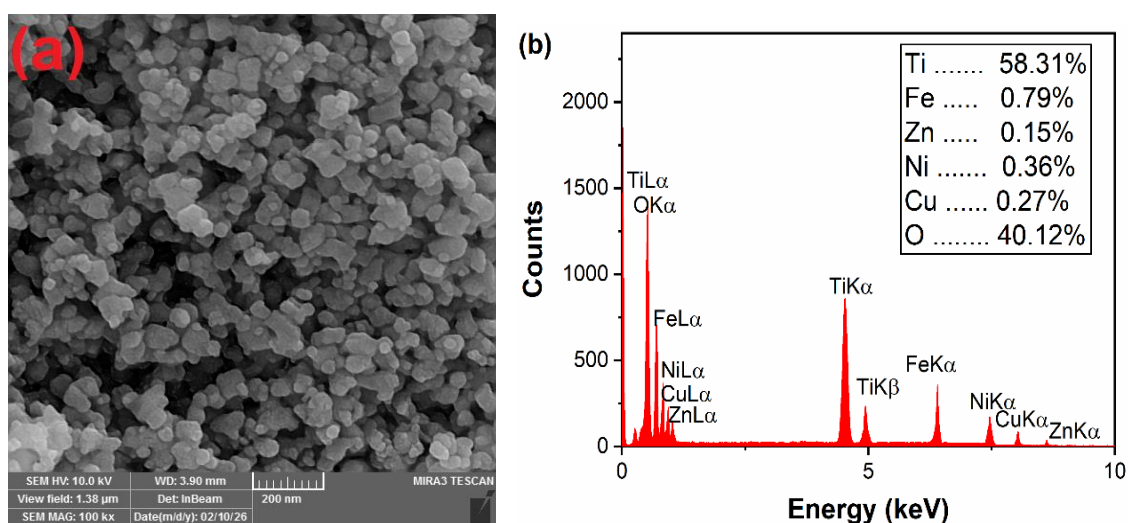


Fig. 2. SEM image (a) and EDX spectrum (b) for the $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite.

To study the photocatalytic efficiency of the $\text{TiO}_2/\text{CFNZO}$ nanocomposite under different experimental conditions, the tests were conducted in the presence of different concentrations of H_2O_2 and various radical scavenger agents. Also, the effect of pH of the AR88 was studied on the photocatalytic activity of the $\text{TiO}_2/\text{CFNZO}$ nanocomposite.

RESULTS AND DISCUSSION

XRD analysis

Fig. 1 represents the XRD patterns for the pure CFNZO and TiO_2 nanoparticles along with $\text{TiO}_2/\text{CFNZO}$ nanocomposite incorporated with different concentration of CFNZO nanoparticles. The XRD pattern for the CFNZO nanoparticles revealed the cubic structure for the prepared nanoparticles (JCPDS: 048-0491). The observed diffraction peaks at $2\theta = 29.45^\circ, 34.73^\circ, 36.61^\circ,$

$42.45^\circ, 52.84^\circ, 56.44^\circ,$ and 62.09° are clearly matched with the aforementioned reference code for the $\text{Cu}_{0.4}\text{Fe}_{1.6}\text{Ni}_{0.65}\text{Zn}_{0.35}\text{O}_4$ compound. As for TiO_2 nanoparticles, the anatase structure was confirmed for the prepared TiO_2 nanoparticles (JCPDS: 04-0477), showing the peaks at $2\theta = 25.34^\circ, 36.86^\circ, 37.78^\circ, 38.49^\circ, 48.11^\circ, 53.91^\circ, 55.12^\circ, 62.11^\circ, 62.69^\circ, 68.57^\circ, 70.31^\circ, 75.08^\circ,$ and 76.11° . The prepared nanocomposite mainly shows the reflections belonged to the TiO_2 phase. The diffraction peaks at around $29.4^\circ, 34.7^\circ,$ and 42.4° (marked with *) are attributed to the incorporated CFNZO nanoparticles. As the concentration of CFNZO incorporated into the TiO_2 matrix increased, the intensity of the associated peaks also increased.

SEM image

The SEM image of the $\text{TiO}_2/2\text{-CFNZO}$

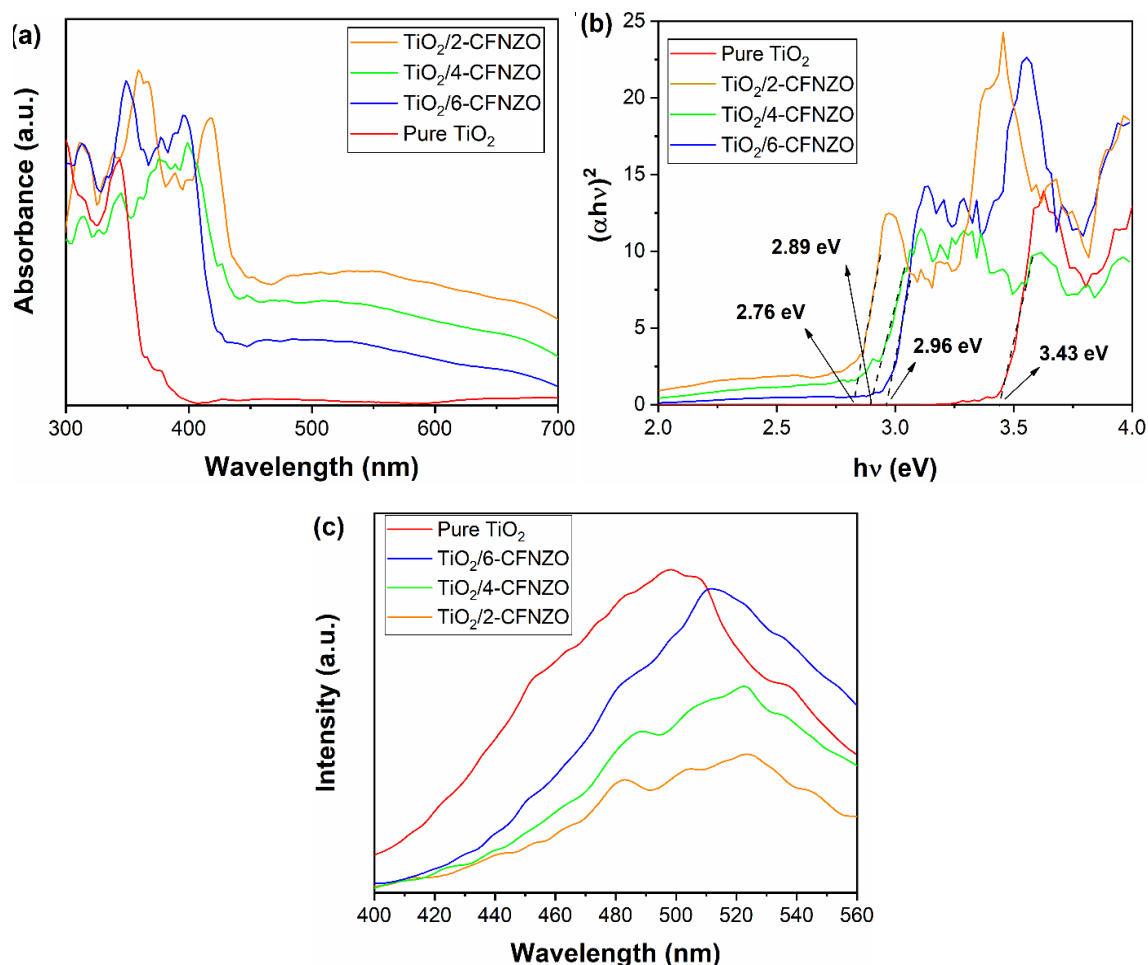


Fig. 3. DRS spectra (a), Tauc plots (b), and PL spectra (c) for the different prepared $\text{TiO}_2/\text{CFNZO}$ nanocomposites.

nanocomposite was shown in Fig. 2a, exhibiting the semi-spherical nanoparticles with the size in the range 50-100 nm. In addition, the composition and microanalysis of the nanocomposite were carried out using energy dispersive X-ray (EDX) spectroscopy, shown in Fig. 2b. As depicted, the nanocomposite is consisted of Ti (58.31%), Fe (0.79%), Zn (0.15%), Ni (0.36%), Cu (0.27%), and O (40.12%).

DRS and PL analyses

Optical properties of the $\text{TiO}_2/\text{CFNZO}$ nanocomposite were studied using DRS and PL spectroscopy. Fig. 3a exhibits the absorption of the nanocomposite, revealing the $\text{TiO}_2/2\text{-CFNZO}$ possesses the higher absorption in the visible light region (400-700 nm). However, no enhancement in the absorption was observed by incorporation of CFNZO by 4 wt% and 6 wt%. This result confirmed that the 2% of the CFNZO nanoparticles effectively contribute to enhance the separation of charge carriers, thereby $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite shows the higher visible light photoactivity. Additionally, the optical band gaps were determined for the nanocomposites as 2.76, 2.89, and 2.96 eV for the $\text{TiO}_2/2\text{-CFNZO}$,

$\text{TiO}_2/4\text{-CFNZO}$, and $\text{TiO}_2/6\text{-CFNZO}$, respectively. In comparison to the prepared nanocomposite, pure TiO_2 has the band gap of 3.43 eV.

For further confirmation of the potential of CFNZO nanoparticles for the enhancement of the photocatalytic activity of TiO_2 , PL analysis was performed to study the separation efficiency of the electron/hole pairs. The results revealed that the incorporation of 2% of CFNZO nanoparticles led to the significant decrease of the PL intensity, implying that the suppressed radiative recombination of the electron hole pairs. However, PL intensity was not positively affected by more amounts of the CFNZO nanoparticles.

Photocatalytic tests

The Photocatalytic performance of the different prepared $\text{TiO}_2/\text{CFNZO}$ nanocomposites (0.04 g) was studied by the visible-light removal of AR88 solution. Fig. 4 shows the performance of the $\text{TiO}_2/\text{CFNZO}$ nanocomposite for the AR88 degradation under 120 min the visible light irradiation. As seen, the $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite represents the optimal performance (90.7%) of the photocatalytic activity, which is attributed to the enhanced visible-light absorption ability. By comparison, the

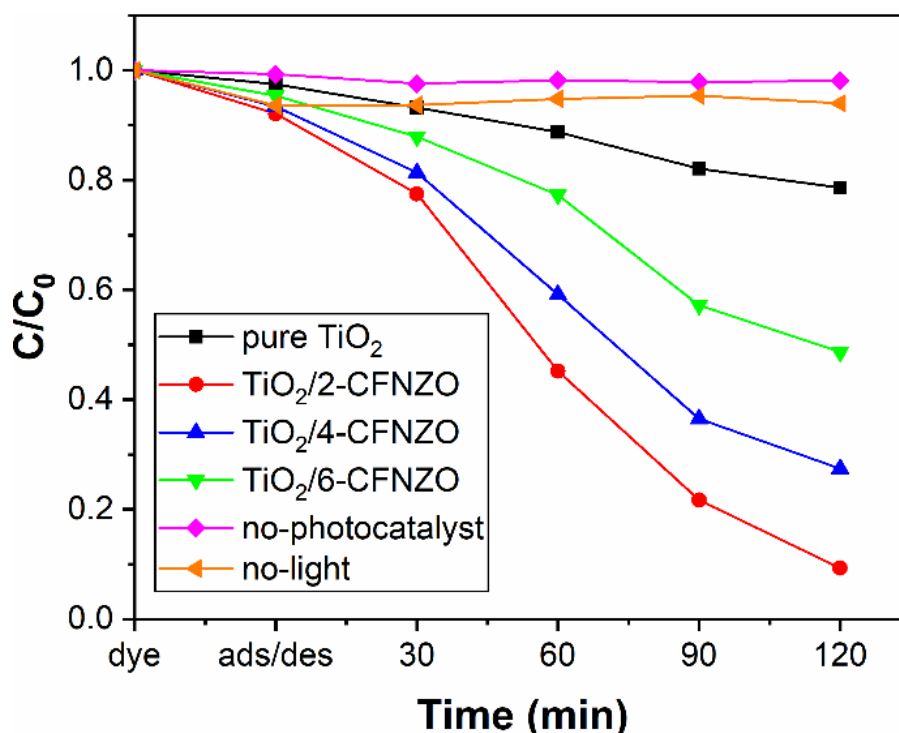


Fig. 4. Photocatalytic degradation of AR88 using different $\text{TiO}_2/\text{CFNZO}$ nanocomposites.

$\text{TiO}_2/4\text{-CFNZO}$ and $\text{TiO}_2/6\text{-CFNZO}$ nanocomposites showed 72.6% and 51.3% of the AR88 degradation during the same irradiation time, respectively. The pure TiO_2 was able to degrade the AR88 solution by only 21.4%. Additionally, the degradation yield of the AR88 was insignificant under no catalyst condition, revealing that the AR88 is stable under the visible light irradiation and represents negligible degradation in the absence of the $\text{TiO}_2/\text{CFNZO}$ nanocomposite. Similarly, when no visible

light source was used, the degradation of AR88 was too low, which is confirmed that the reported degradation efficiency of AR88 solution can be truly attributed to the photocatalytic reaction over the $\text{TiO}_2/\text{CFNZO}$ nanocomposite.

Photocatalyst amount

In order to study the optimal amount of the $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite to attain the highest photocatalytic yield, different amounts of the

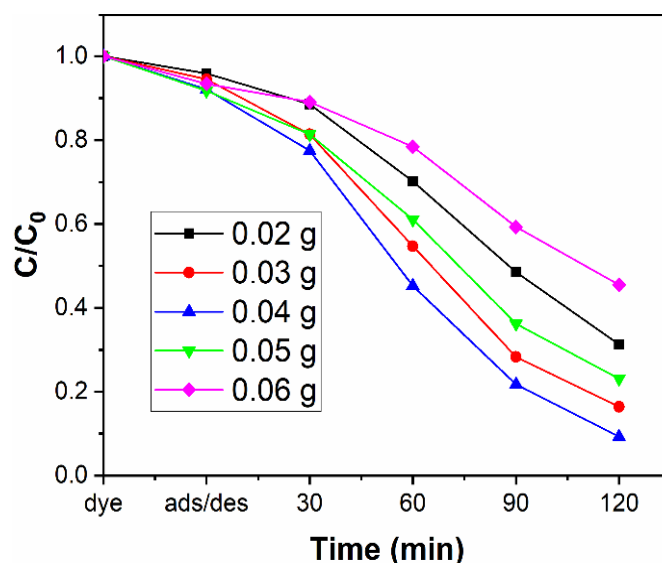


Fig. 5. Effect of different amount of $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite on degradation level of the AR88 solution.

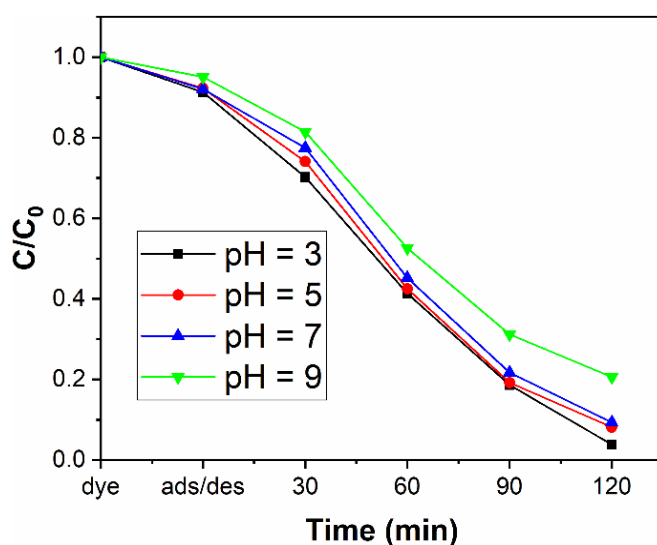


Fig. 6. Effect of AR88 solution pH on the photocatalytic performance of $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite.

nanocomposite (0.02, 0.03, 0.04, 0.05, and 0.06 g) were loaded and the degradation level of the AR88 was carefully monitored, as shown in Fig. 5. The photocatalytic degradation of AR88 solution reached to the highest level using 0.04 g of the $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite. As can be seen, the exceeded amount (0.05 and 0.06 g) caused to prominent decrement in the photocatalytic yield. More amount of the loaded photocatalyst, the more active site is. However, when overuse of the photocatalyst leads to a relatively turbid solution, impeding the light beams to penetrate into the solution effectively, and thereby activate the photocatalyst [23-25].

pH of AR88

Fig. 6 shows the effect of pH of the AR88 on the photocatalytic performance of the $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite. As can be seen, the nanocomposite shows the better result at lower pH values, so that the degradation yield reached to 96.2% at pH of 3. However, the photocatalytic efficiency of the $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite gradually decreased by increasing the pH values to 9. At pH of 9, the photocatalytic activity of the nanocomposite approached 79.4%. These observations can be justified by concluding that

anionic AR88 molecules increasingly adsorb on the positively charged surface of photocatalyst [26-29]. Consequently, AR88 is more oxidized under acidic conditions.

H₂O₂ concentration

The photocatalytic reaction is largely affected by the oxidant agents, due to contribution of oxidants to photocatalytically formation of radicals— $\cdot\text{OH}$ or $\cdot\text{O}_2^-$ [30]. In this work, H_2O_2 was used to elevate the degradation yield of the AR88 solution over the $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite. Fig. 7 exhibits the impact of the addition of different amounts of H_2O_2 (0.02 M) on the photocatalytic activity of the $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite. The H_2O_2 had beneficial effect on the photocatalytic activity of the $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite. More than 98.3% of the AR88 was removed with the addition of 2 mL of H_2O_2 (0.02 M). More amounts of the H_2O_2 (4 mL) led to the dramatic decrease in the photocatalytic activity, which is attributed to the suppression of the formation of radicals during the photocatalytic reactions [31].

Scavenger agents

The photocatalytic experiments were also carried out in the presence of different radical

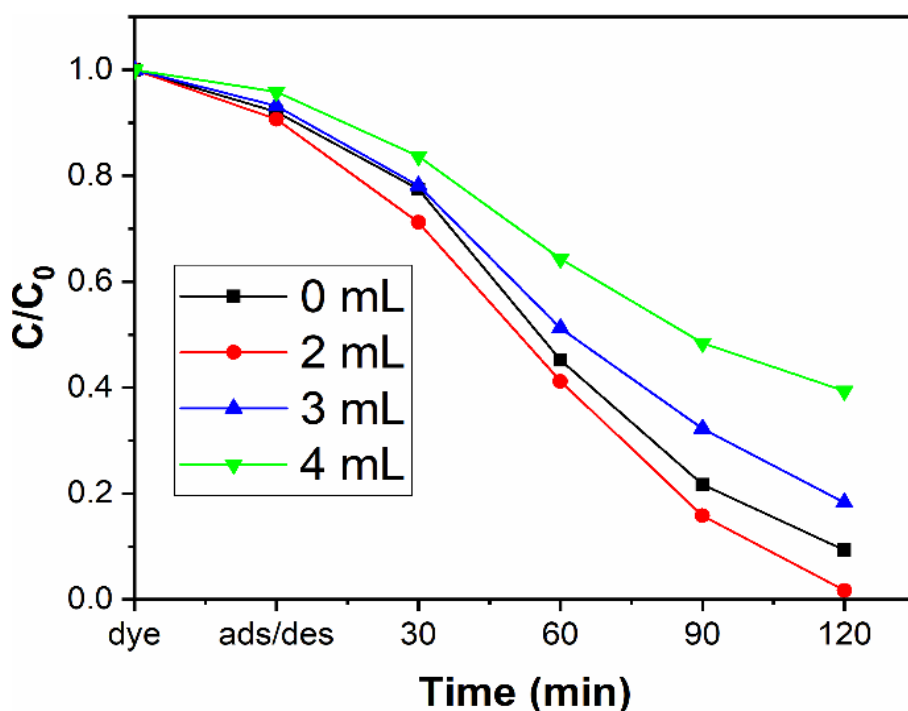


Fig. 7. Effect of H_2O_2 concentration on the photocatalytic performance of $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite.

scavenger agents, including disodium salt of ethylenediaminetetraacetic acid (EDTA), isopropanol, and ascorbic acid for hindering the photo-generated holes (h^+), hydroxyl radicals ($\cdot\text{OH}$), and superoxide radicals ($\cdot\text{O}_2^-$), respectively [32-34]. Fig. 8 shows the effects of the addition of (2 mM) different radical scavenger agents on the photocatalytic degradation of AR88 solution. The results showed the photocatalytic activity of the $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite decreased as EDTA and isopropanol were added, confirming that the photocatalytic degradation of AR88 over the $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite dominantly performed by $\cdot\text{OH}$ radicals and photo-generated h^+ .

Reusability tests

The stability of the photocatalyst for multiple uses in an important feature of a practical photocatalyst [35]. In order to study the reusability and structural stability of the prepared $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite, the photocatalytic degradation reactions were conducted under 7 successive cycles. All the reaction were carried out using the constant amount of the nanocomposite (0.04 g), which was recovered after each reaction cycle by centrifugation at 7000 rpm for 5 min, washed using ethanol solution (20% V/V) and then dried at 90°C for 4 h. Fig. 9 shows the degradation efficiency of AR88 under 7 successive reactions. As shown, the $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite

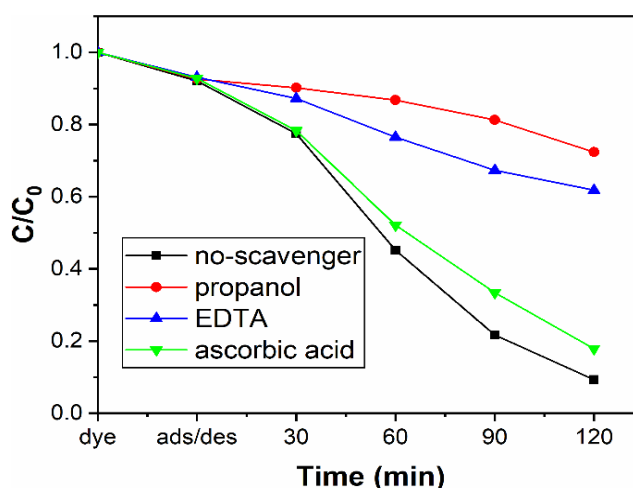


Fig. 8. Radical scavenging experiments results for the photocatalytic degradation of AR88 using $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite.

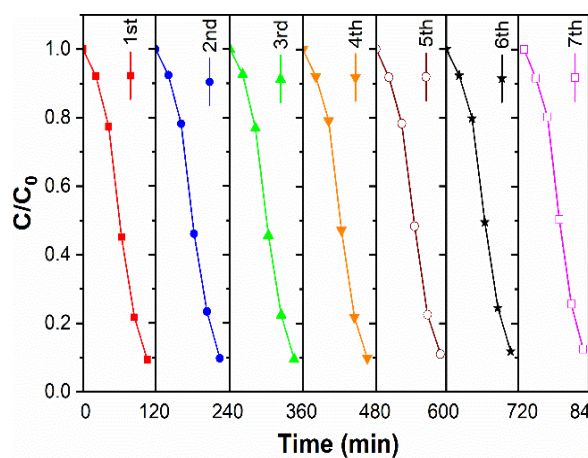


Fig. 9. AR88 degradation results over 7 consecutive reaction cycles using $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite.

possesses the superior reusability without significant decrease in the degradation efficiency over 7 reaction cycles. After 7th reaction, the decrease in the photocatalytic activity was only 3.28%.

CONCLUSION

In summary, we have prepared $\text{TiO}_2/\text{Cu}_{0.4}\text{Fe}_{1.6}\text{Ni}_{0.65}\text{Zn}_{0.35}\text{O}_4$ nanocomposite using two steps reaction, including co-precipitation and sol-gel methods. The characterization of the nanocomposite was performed to investigate the ability of the nanocomposite to function as the visible-light photocatalyst. The band gap values of the $\text{TiO}_2/\text{Cu}_{0.4}\text{Fe}_{1.6}\text{Ni}_{0.65}\text{Zn}_{0.35}\text{O}_4$ nanocomposites including different concentrations of the CFNZO nanoparticles (2%, 4%, and 6%) were determined to be 2.76, 2.89, and 2.96 eV, respectively. Accordingly, the $\text{TiO}_2/2\text{-CFNZO}$ nanocomposite was used to study the visible-light removal of AR88 within 120 min photocatalytic reaction. The degradation yield was obtained about 90.7% using 0.04 g of the prepared nanocomposite. Additionally, after careful examination, it was found that the prepared nanocomposite showed the better photoactivity in acidic conditions and in the presence of H_2O_2 (2 mL, 0.02 M). The radical scavenging experiments also exhibited that the degradation of AR88 was accomplished using hydroxyl radicals and photo-generated holes. Furthermore, the prepared nanocomposite showed the superior photocatalytic activity over 7 consecutive reaction cycles, without significant loss of photocatalytic performance.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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