Photocatalytic Activity of Ti-SBA-15/C3N4 for Degradation of 2,4-Dichlorophenoxyacetic Acid in Water under Visible Light

Hoa T. T. Duong, Mai T. P. Duong, Oanh K. Nguyen, Son T. Le, Long V. Dang, Binh T. Nguyen, and Dang V. Do

Faculty of Chemistry, VNU University of Science, Vietnam National University, Ha Noi 19 Le Thanh Tong Hoan Kiem, Hanoi 100000, Vietnam

Correspondence should be addressed to Dang V. Do; dangdovan@hus.edu.vn

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In the present study, the photocatalytic activity of Ti-SBA-15/C3N4 catalysts was investigated to degrade 2,4-Dichlorophenoxyacetic acid (2,4-D) herbicides in water under visible light irradiation. The catalysts were synthesized via a simple hydrothermal method and characterized by various analytical techniques, including SAXS, N2 adsorption-desorption isotherms, Zeta potential, PL, FT-IR, XRF, TGA, and UV-DRS. Our study indicated that the 2.5Ti-SBA-15/C3N4 had higher efficiency in the degradation of 2,4-D than Ti-SBA-15 and C3N4. The decomposition of 2,4-D reached 60% under 180 minutes of visible light irradiation at room temperature on 2.5Ti-SBA-15/C3N4. Moreover, the degradation of 2,4-D on Ti-SBA-15/C3N4 was pseudo-first-order kinetics with the highest rate constant (0.00484 min⁻¹), which was much higher than that obtained for other photocatalysts reported recently. Furthermore, the catalyst can be reused at least two times for photodegradation of 2,4-D solution under visible light irradiation within a slight decrease in catalytic activity.

1. Introduction

Last century, 2,4-Dichlorophenoxyacetic acid (2,4-D) was one of the most generally used herbicides in agricultural producing countries [1, 2]. 2,4-D has primarily been used in agriculture to control weeds in corn and grain, forestry, and lawn care practices [3]. Still, its harmful effects are severe, such as harsh contaminants of superficial and ground waters due to its slow degradation by biological processes, affecting amphibious life in water bodies, causing chromosomal aberrations in human lymphocytes [4, 5]. Therefore, the development of methods to remove wastewater contaminants has gained considerable interest in commercial and academic worldwide. However, most current techniques for the degradation of 2,4-D, such as photo Fenton oxidation and microbial degradation, have to be carried out in a complicated process, producing hazardous by-products or expensive costs [6–8].

Recently, photocatalysis has been a promising advanced water treatment process to replace or support current methods [9]. Titanium-based catalyst has been considered one of the most popular photocatalysts to degrade persistent organic pollutants such as 2,4-D and lindane. Among those, Ti-SBA-15 has been widely used as a catalyst, photocatalyst, and catalyst support for other materials because of its highly ordered and uniform mesoporous channels, thick silica walls, and large surface area [10]. However, several studies indicated that Ti-SBA-15 had limited practical application under visible light conditions because it only absorbed UV light [11, 12]. On the other hand, graphitic carbon nitride (g-C3N4), a metal-free material with a low electronic bandgap falling in the visible light range of the solar spectrum, has some advantages in photocatalytic applications such as chemically stability under ambient conditions, flexible optical, and non-toxic [13, 14]. g-C3N4 was also modified by various semiconductors (e.g., Ag2S, CdS, Bi2O3I2, Ag3PO4, Ni1Ca@Ni, ZnO, Bi2O3–V2O5) or element doping (N, P, S) to improve its photocatalytic degradation of dye or hydro evolution [15–19]. To the best of our insight, there are no
To develop more visible light-efficient catalysts, it is crucial to design photocatalytic systems that are able to operate effectively [20]. Herein, Ti-SBA-15 loading with carbon nitride was prepared and studied for an efficient photocatalyst for the degradation 2,4-D under visible light at neutral pH. The relationship between absorption amount and the photodegradation of 2,4-D on Ti-SBA-15/C3N4 was discussed in detail. Furthermore, the 2,4-D degradation processes on Ti-SBA-15/C3N4 have also exchanged views about the kinetics and mechanism in this study.

3. Results and Discussion

3.1. Characterization of xTi-SBA-15 and xTi-SBA-15/C3N4. Figure 1 shows the SAXS patterns of xTi-SBA-15 and samples modified with C3N4. Ti-SBA-15 Figure 1(a) has a sharp diffraction peak at $q = 0.67 \text{nm}^{-1}$ ($2 \theta = 0.94^\circ$) due to (100) reflections of a hexagonal mesoporous lattice. In addition, small peaks were also observed at $1 \text{nm}^{-1} < q < 1.5 \text{nm}^{-1}$ ($4 \theta < 2 \theta < 2.1^\circ$), corresponding to (110), (200) reflections of the mesopores in this pattern. These results indicate the formation of a hexagonally packed porous structure. For xTi-SBA-15, all features for hexagonal lattice were observed without their slight decrease in peak intensities. The respective q values were also decreased from 0.67 nm$^{-1}$ to 0.621 nm$^{-1}$ along with the increase of the Ti amount. The unit cell parameter was 10.8 nm for pure SBA-15 and increased in the presence of Ti in the porous structure. The difference in the unit cell parameter indicates the larger size of the Ti heteroatom incorporated into SBA-15. It suggested that the long-range order of the mesoporous structure of SBA-15 has remained [22]. Moreover, the SAXS patterns of all xTi-SBA-15/C3N4 also show hexagonal
diffraction peaks due to reflections of (100), (110), and (200) without a slight decrease in intensity. Figure 1(b), indicating that the structure of these materials has remained after modification. The XRD of xTi-SBA-15/C₃N₄ (x=0, 2.5) were also measured at wide-angle and shown in Figure 2(a). The results showed that both materials presented a broad peak at 2θ = 27.5°, corresponding to the (002) plane of C₃N₄ within the decreasing intensity, suggesting that the structure of C₃N₄ has also remained after modification by xTi-SBA-15.

The N₂ adsorption-desorption measurements also confirmed the information on the mesoporous structure Figure 2(b). The typical type IV isotherms were observed with large H1 hysteresis loops at a high relative pressure of 0.6–0.8, corresponding to capillary condensation of nitrogen in the uniform mesopores. Table 1 and Figure 2(d) show the pore structure character parameters of the synthesized materials. The surface area, pore-volume, and pore size of Ti-SBA-15 increase slightly compared to SBA-15. It could be due to Ti atoms replacing Si atoms and expanding the SBA-15 structure [23, 24].

Electronic and optical properties are of fundamental importance to determine the photoactivity of a catalyst. UV-DRS spectroscopy was applied for characterizing the optical properties of the catalysts.

The weak peak is observed at 256 nm for all the samples xTi-SBA-15/C₃N₄ and pure C₃N₄, attributed to the aromatic ring’s p → π* Figure 3(a). Besides, the intense peak at 380 nm corresponding to the n → π* transitions is caused by electron transfer from nonbonding nitrogen orbital to an aromatic anti-bonding orbital. The absorption band of xTi-SBA-15 is slightly toward long-wavelength than C₃N₄, as well as an increase in the absorption of visible light. The Tauc plots Figure 3(b) show the bandgap of xTi-SBA-15 (2.73 eV, 2.72 eV, and 2.72 eV, respectively) slightly decreased in comparison with pure C₃N₄ (2.74 eV). The C₃N₄ bandgap value corresponds to -1.13 eV, 1.59 eV of the conduction band (CB), and valence band (VB). It is hard to confirm the CB and VB of Ti-SBA-15; however, it is assumed that they were near the TiO₂ value (-0.5 eV, 2.7 eV of CB and VB, respectively). From these results, it could be demonstrated that the promoted xTi-SBA-15/C₃N₄ materials are more active than C₃N₄ under visible light irradiation.

Photoluminescence (PL) spectra were also measured to analyze photogenerated electron-hole pairs’ migration, transfer, and recombination process in the photocatalysts (Figure 4). The emission peak of all materials was at around 470 nm, indicating the structure of C₃N₄ has remained after modification. The peak intensity of xTi-SBA-15/C₃N₄ decreases along with the increase of Ti amount. It is noticed that the PL intensity of 2.5Ti-SBA-15/C₃N₄ was the lowest, corresponding to lower recombination of electron-hole pair.

The FT-IR spectra of these materials are shown in Figure 5. The results indicated that the carbon nitride replicas show many peaks due to carbon nitride networks’ formation by condensing precursors, which can be assigned for C–N heterocycle at 1426 cm⁻¹, 1320 cm⁻¹, and 1243 cm⁻¹ [25]. The band at 1060 cm⁻¹ corresponds to Si–O–Si's
asymmetric stretching vibration, and the peaks at 810 cm$^{-1}$ can be allocated to the symmetric stretching and distortion forms of Si–O–Si [26]. Additionally, the band at 960 cm$^{-1}$ is generally an identity of Si–O–Ti bonds’ existence, as seen in Figure 5.

The thermal gravimetric analysis (TGA) was conducted in the air to quantify the relative amount of 2.5Ti-SBA-15 in 2.5Ti-SBA-15/C$_3$N$_4$ materials. The C$_3$N$_4$ was still stable at 600$^\circ$C before complete decomposition at 700$^\circ$C. However, the 2.5Ti-SBA-15/C$_3$N$_4$ lost 30% of weight at 200$^\circ$C,

| Sample          | Surface area (m$^2$g$^{-1}$) | Pore volume (cm$^3$g$^{-1}$) | Pore size (nm) |
|-----------------|------------------------------|-----------------------------|---------------|
| SBA-15          | 695                          | 1.01                        | 5.33          |
| 1Ti-SBA-15      | 811                          | 1.13                        | 5.29          |
| 2.5Ti-SBA-15    | 986                          | 1.35                        | 5.4           |
| 5Ti-SBA-15      | 1011                         | 1.37                        | 5.37          |
| C$_3$N$_4$      | 26                           | -                           | -             |
| SBA-15/C$_3$N$_4$ | 117                        | -                           | -             |
| 2.5Ti-SBA-15/C$_3$N$_4$ | 123                  | -                           | -             |

Figure 2: (a) XRD patterns; (b) and (c) Nitrogen sorption isotherms; (d) Pore size distributions of synthesized materials.
corresponding to the water loss in the pore of 2.5Ti-SBA-15. Then C3N4 in the composite decomposed entirely at high temperature (>600°C), and 7.5% weight corresponds to the remaining 2.5Ti-SBA-15 (Figure 6).

3.2. Photodegradation of 2,4-D over Ti-SBA-15/C3N4. The photocatalytic activity of the catalysts was investigated in the photodegradation of 2,4-D in water under visible light irradiation. SBA-15 is known to be inert for 2,4-D degradation under UV light or visible light irradiation, even in the sight of O2. By contrast, Ti-SBA-15 is reactive and can promote the degradation of 2,4-D in water under UV light irradiation. However, the reaction yield is relatively low, less than 10% compared to C3N4 (42%) (the data not shown).

The results in Figure 7 demonstrated that all xTi-SBA-15/C3N4 materials have higher degradation of 2,4-D compared to C3N4 under visible light irradiation coincident with UV-DRS and PL results. To find a suitable way to explain this phenomenon, the zeta potentials of Ti-SBA-15, SBA-15/C3N4, and Ti-SBA-15/C3N4 were -20.7 mV, -20.1 mV, and -13.6 mV, respectively, at pH = 7. The experimental results displayed that the 2,4-D adsorption depends on the amount of Ti in the Ti-SBA-15 Figure 7(a). There is a slight increase in absorption amount from 22% to 35%, together with the raising of Ti amount. It coincides with the result of the N2
adsorption-desorption and zeta potential. There is an increase in the photoreaction yield, followed by a significant rise from 0 to 2.5% Ti in materials. The increase in absorption amount also evidenced it. It suggests that the absorption amount might be promotive of the degradation of 2,4-D under visible light irradiation. The 2.5Ti-SBA-15/C3N4 exhibited the highest photodegradation rate of 2,4-D under visible light up to 60% for 180 minutes of irradiation. In contrast, the highest amount of Ti (in 5Ti-SBA-15/C3N4) decreased the photodegradation of 2,4-D under visible light irradiation corresponding to UV-DRS and PL data Figure 7(b). There may exist two possible reasons: (i) the Ti-SBA-15 acts as the role of absorber for the 2,4-D, while g-C3N4 absorbs visible light to release electrons in CB (−1.13 eV) and hole (h+) in VB; (ii) the electrons transferred to CB of Ti-SBA-15 (−0.5 eV), which is redox O2 to O2· radical (E(O2/O2·) = −0.16 V) [27] and OH· radical (E(O2 OH·) = 1.27 V) [28]. The O2·, OH· radical or hole (h+) of heterojunction of Ti-SBA-15/C3N4 can oxidize 2,4-D to degradation products like CO2 and water, as presented in Figure 8.

To further study the reaction kinetics of the 2,4-D photodegradation by xTi-SBA-15/C3N4, the photodegradation data were adapted by implementing a pseudo-first-order model as given by \[ \ln \left( \frac{C}{C_0} \right) = -kt \], where \( k \) is the
The apparent first-order rate constant (min$^{-1}$); $C_0$ is the initial concentration at $t = 0$, and $C$ is the concentration at time $t$ of the 2,4-D solution. Figure 9 shows the linear relationship between $\ln(C_0/C)$ and the irradiation time of photodegradation of 2,4-D on the catalysts. It was clearly observed that the photodegradation curve in all cases matched smoothly with pseudo-first-order kinetics. In addition, the apparent rate constants $k$ of all the catalysts were estimated and decorated in line with the model (Figure 9). The rate constants $k$ values were significantly increased with the rise of Ti loading. The 2.5Ti-SBA-15/C$_3$N$_4$ catalyst displayed the highest rate constant (0.00484 min$^{-1}$) for 2,4-D degradation, which was much higher than that obtained for other photocatalysts reported recently as ZnO–FeY [29], PdO/Al$_2$O$_3$–Nd$_2$O$_3$ [30], chitosan–TiO$_2$ beads [31], Fe/TiO$_2$ [32] or photo-Fenton process [33].

To optimize the effect of 2,5Ti-SBA-15 in the composite for 2,4-D photodegradation, numerous composites with different amounts of 2,5Ti-SBA-15 were also prepared. Figure 10 shows the decomposition of 2,4-D, and Figure 11 displays the relationship between $\ln(C_0/C)$ and the irradiation time of photodegradation depending on the amount of 2,5Ti-SBA-15 in target materials. The photodegradation of 2,4-D under visible light irradiation is increased from 50% ($k = 0.00397$ min$^{-1}$) to 60% ($k = 0.00484$ min$^{-1}$), coincident with the rise of 2,5Ti-SBA-15 from 2% to 7.5%. However,
when 2.5Ti-SBA-15 increases up to 10% wt, the photodegradation of 2,4-D slightly decreases, whereas the absorption amount was highest. The decreased absorption of visible light may cause it.

Finally, the recycling ability of the 2.5Ti-SBA-15/C₃N₄ catalyst was investigated for degradation of the 2,4-D solution under visible light irradiation. The experiments were also carried out using the same procedure without taking the liquid from the mixture reaction. After 180 minutes, the solid was collected by centrifugation and washed with water several times. Then, the solid was dried at 100°C and used for the next reaction. The results showed that 2.5Ti-SBA-15/C₃N₄ is recycled at least two times with slight losing catalytic activity. The decrease would be ascribed to the loss of catalyst during experiments (Figure 12). From these results, it is demonstrated that the photocatalyst is robust and stable.

**4. Conclusions**

In an effort to develop photocatalysts, this study reported the catalytic activity of Ti-SBA-15/C₃N₄ for the degradation of 2,4-D under visible light irradiation. These synthesized materials were characterized by various analytical methods such as SASX, XRD, UV-DRS, N₂ adsorption-desorption isotherms, Zeta potential, XRF, PL, TGA, and FT-IR. The photodegradation of 2,4-D over Ti-SBA-15/C₃N₄ is much higher than pure Ti-SBA-15, SBA-15/C₃N₄, or C₃N₄. The reaction proceeded to give excellent efficiency on 2.5Ti-SBA-15/C₃N₄ with 60% degradation 2,4-D under visible light at room temperature for 180 minutes. The photodegradation of 2,4-D was pseudo-first-order kinetic with the highest constant \( k = 0.00484 \text{ min}^{-1} \), much higher than that obtained for other photocatalysts reported recently. In addition, the catalyst can be reused at least two times within a slight decrease of photocatalytic activities. It was realized that Ti-SBA-15 was an absorber and Electron migration exists in the heterojunction of Ti-SBA-15/C₃N₄ to enhance the photocatalytic activity of the catalyst. Since a good correlation was established between the adsorption capacity and amount of Ti in Ti-SBA-15/C₃N₄, 2.5Ti-SBA-15 was determined to improve the adsorption property for 90 minutes.

**Data Availability**

The data used to support the findings of this study are included in the article.

**Conflicts of Interest**

The authors declare that there are no conflicts of interest.

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