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Enhanced visible light photocatalytic activity of SDBS/C/Nd/TiO_2 synthesized by using anionic surfactant SDBS as template agent

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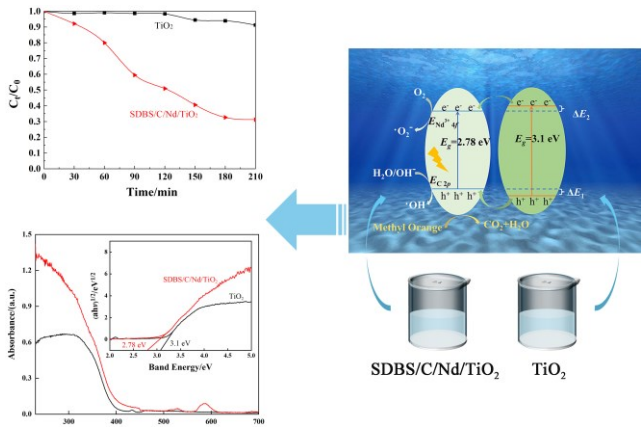
Graphical abstract



Abstract

The catalyst SDBS/C/Nd/TiO_2 was synthesized by sol-gel method using TiO_2 co-doped with C and Nd and anionic surfactant SDBS as template agent. The prepared photocatalysts were characterized by X-ray diffraction (XRD), scanning electron (SEM), X-ray photoelectron spectrometer (XPS) and UV-vis diffuse reflectance spectroscopy (UV-vis DRS) and so on. Experimental results show that the crystal structure of the catalytic material is anatase type. Since doped elements change the energy band structure and electronic structure of TiO_2, photogenerated electrons and holes can transition through the intermediate energy level, resulting in a decrease in the required excitation energy, thereby increasing the distance of its absorption edge moving toward the long wave direction, and meanwhile improving the utilization rate of visible light. The bandgap energy of the catalyst SDBS/C/Nd/TiO_2 was 2.78 eV. Compared to pure TiO_2 (3.1 eV), the band gap is reduced by 0.32eV. The photocatalytic experiments show that the degradation rate of the catalyst SDBS/C/Nd/TiO_2 for methyl orange solution can reach 67%, and the catalytic performance is much improved than that of pure TiO_2.

Keywords: Sol-gel method, anionic surfactant, catalytic material, energy level, catalytic performance

1. Introduction

The excessive manipulation and use of resources and the deep pollution of nature pose a certain threat to the environment on which human beings depend for survival. Photocatalysis has shown a wide application prospect in the field of remediation of environmental pollution, thus making this technology receive wide attention and become a hot spot of current research (Xie *et al.* 2014; Sudarsanam *et al.* 2020; Xia *et al.* 2023). In general, the photocatalytic materials have many advantages, such as the low toxicity, cheap cost, mild conditions, good chemical stability and strong oxidation ability (Yu *et al.* 2017; Krylov *et al.* 2021), but they only have certain absorption performance under the condition of ultraviolet light, which results in the decrease of solar energy utilization (Xiao *et al.* 2016). This is one of the disadvantages that has been restricting the improvement of photocatalytic activity. Therefore, how to modify the photocatalytic materials and extend the field of light response to the visible region, so that it can be applied to real production and life, has become the focus of in-depth research (Kuerbanjiang *et al.* 2013).

A more promising photocatalyst under current conditions is TiO_2 (Verbruggen, 2015). In order to improve the photocatalytic efficiency of TiO_2 in the visible light range, people has adopted a series of measures, such as: heterostructure (Liu *et al.* 2012), dye sensitized (Zhao *et al.* 2023) and ions doped (Yin *et al.* 2005; Dwiyanna *et al.* 2021), in which the ions doped is an easy operation and effective method. The ions doped include nonmetal, metal ion, inert metal and rare earth metal doped (Kader *et al.* 2022; Kunarti *et al.* 2020; Jin *et al.* 2023; Vishwakarma *et al.* 2023). The co-doped of two or more ions can significantly improve the photocatalytic activity due to the synergistic effect of ions (Sirivallop *et al.* 2020; Wang *et al.* 2022; Chen *et al.* 2017). Among them, the rare earth metal ions have an unfilled 4*f* layer for transition. Due to such special structure, they are of great help to expand the range of absorption spectrum, more and more scholars have studied them, in which Nd element has received a lot of attentions as the rare earth element.

Rengaraj (Rengaraj *et al.* 2007) pointed out that Nd doped TiO_2 can capture electron centers, provide more charge carriers and effectively separate electrons and holes on the surface of TiO_2 through redox reaction. After decades of research and development, semiconductor photocatalytic technology has been applied in the fields of solar energy utilization, environmental improvement and medical health (Chen *et al.* 2010), but still has some problems: such as, the narrow spectral response and the low quantum efficiency result in a certain distance in its industrial production.

In the process of material synthesis, the addition of surfactant plays an important role in regulating the morphology of catalyst. Sodium dodecyl benzene sulfonate (SDBS) is a multifunctional chemical whose applications cover many different fields. Its surface activity and dispersion ability make it one of the indispensable ingredients in a variety of products and industrial processes. Whether used in detergent manufacturing, pharmaceutical preparations, laboratory research or other industrial applications, SDBS plays an important role. In addition, SDBS can also be used as a structure-oriented agent when crystal growth is performed by hydrothermal or solvothermal methods. At the same time, the morphology and particle size of the material can be changed by controlling the amount of SDBS (Zhang *et al.* 2023; Si *et al.* 2018).

In this paper, SDBS/C/Nd/TiO_2 catalyst was designed and synthesized by sol-gel method (Marzec *et al.* 2016; Li *et al.* 2015) using SDBS as template agent. By adding the anionic surfactant SBDS, the particle size of the catalyst can be uniformly dispersed, the particle size is consistent, and the specific surface area and pore size of the catalyst can be expanded. The photocatalytic properties of the prepared catalysts were tested with methyl orange solution as the probe, and the photocatalytic mechanism was studied by combining experiment results. The results show that the prepared SDBS/C/Nd/TiO_2 achieves a significant enhancement in photocatalytic performance compared with the pure TiO_2.

2. Materials and methods

2.1. Materials

Butyl titanate (AR, 98%) and glucose (AR, 98%) were obtained from Tianjin Kemi Ou Chemical Reagent Co., LTD, sodium dodecyl benzoate (AR, 90%) was obtained from Zhengzhou Yongxing Chemical Co., LTD, anhydrous ethanol (AR, 75%), neodymium nitrate (AR, 99%), glacial acetic acid (AR, 36%) and nitric acid (AR, 65%-68%) were obtained from Tianjin Kaitong Chemical reagent Co., LTD.

2.2. Synthesis

2.2.1. TiO_2 catalyst

TiO_2 catalyst was synthesized by sol-gel method. Firstly, a certain volume of butyl titanate was measured and added to anhydrous ethanol, then, the mixture was stirred for 30 minutes, denoted as liquid A. A certain volume of distilled water and anhydrous ethanol were mixed simultaneously, and dilute nitric acid was used to adjust the pH of the solution, denoted as liquid B. Add the B solution to the A solution drop by drop, and in a certain temperature under stirring for several hours, get wet sol, transferred to the reaction kettle in drum wind drying oven aging 2 d, decompression filtration, 100 ℃ vacuum drying 2 h. Thus, TiO_2 catalyst was successfully prepared.

2.2.2. SDBS/C/Nd/TiO_2 catalyst

SDBS/C/Nd/TiO_2 catalyst was prepared by sol-gel method using SDBS as template. A certain volume of butyl titanate was measured and added to anhydrous ethanol, then, the mixture was stirred for 30 minutes, denoted as liquid A. Anhydrous ethanol, deionized water, glacial acetic acid, glucose, $Nd(NO_3)_3$ and SDBS were measured and added into the beaker to dissolve and dilute nitric acid was used to adjust the pH of the solution, denoted as liquid B. Add the B solution to the A solution drop by drop, and in a certain temperature under stirring for several hours, get wet sol, transferred to the reaction kettle in drum wind drying oven aging 2 d, decompression filtration, 100 ℃ vacuum drying 2 h. Subsequently, the SDBS/C/Nd/TiO_2 catalyst could be obtained.

2.3. Characterization

A Japanese HITACHI s-4300 scanning electron microscopy and Japanese H-7650 transmission electron microscopy were used to observe the catalysts in terms of their dispersion, morphology and particle size; a D8-FOCUS X-ray diffractometer from Germany was used to test the crystal structure; X-ray photoelectron spectroscopy of ESCALAB250Xi from Thermo, USA was used to explore the classification of material elements, the combination of elements and their percentages; the characteristics of absorption peaks were used for qualitative analysis by the TU-1901 UV-visible diffuse reflection; the N_2 adsorption-desorption of the catalysts were measured by Quntachrome Nova Win2 physical adsorption instrument of konta and the specific surfaces and pore volumes of the catalysts were calculated by BET and BJH.

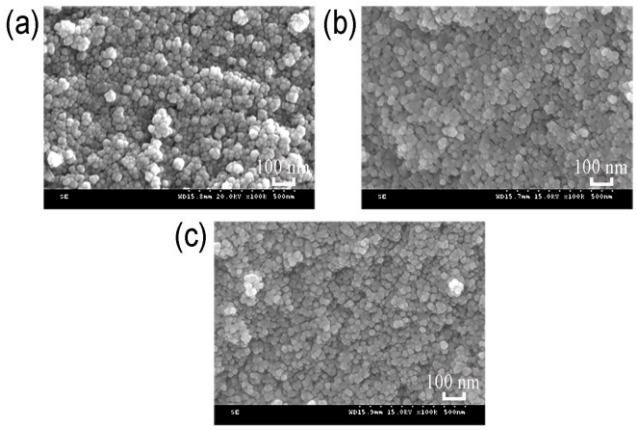


Figure 1. SEM of a) TiO2, b) SDBS and c) SDBS/C/Nd/TiO2

2.4. Photocatalytic performanc

Photocatalytic experiments were carried out by using 1000 W xenon lamp as the simulating light source and using methyl orange solution as the probe. 50 mL of the dye solution was added in the test tube, and 0.1g of different catalysts were weighed in the above solution, respectively, which was put into the light reaction and

stirred for 30 minutes closing the light, the sample was denoted as 0 min. Open the light, and the sample was taken every 30 min. The TU1901 UV-visible spectrophotometer was used to test the absorbance. The decolorization rate was calculated by formula 1, and the performance of dye degradation was compared：

$$\eta=\frac{A_0-A_t}{A_0}\times 100\% \quad (1)$$

Formula 1 represents the formula of decolorization rate, where A_0 represents the absorbance of the dye solution after being stirred for 30 minutes in the dark; A_t is the absorbance of the solution light t min.

3. Results and discussion

3.1. Morphology of catalyst

Scanning electron microscopy (SEM) was used to analyze the morphology of the samples. The SEM images of TiO_2、SDBS and SDBS/C/Nd/TiO_2 are shown in Figure 1a-1c, respectively. It could be seen that all three samples are composed of spherical particles. It is obvious that the addition of anionic surfactant SBDS is conducive to the same surface particle size and uniform dispersion. Compared with pure TiO_2, the surface morphology of TiO_2 was improved by using sodium dodecyl sulfate as a surfactant.

3.2. X-ray diffractometer

The XRD patterns of TiO_2 and SDBS/C/Nd/TiO_2 were shown in Figure 2a. By comparing the standard JCPDF cards, it is obvious that all the strong patterns are in agreement with the pure anatase-type TiO_2. At the same time, no other impurity diffraction peaks appeared, which clearly demonstrate the phase purity.

Table 1. The information for the samples TiO_2 and SDBS/C/Nd/TiO_2

Samples	TiO_2	SDBS/C/Nd/TiO_2
Band gap/eV	3.10	2.78
Specific surface area/m^2g^{-1}	55.22	116.1
Pore volume/cm^3g^{-1}	0.089	0.361

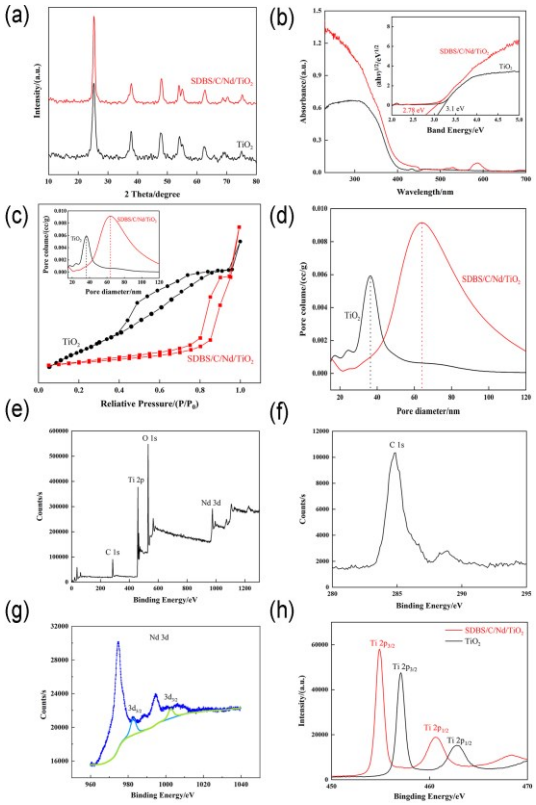


Figure 2. a) XRD spectrum of TiO2 and SDBS/C/Nd/TiO2; b) UV-vis-NIR diffuse reflectance spectrum of TiO2 and SDBS/C/Nd/TiO2; c) and d) the N2 adsorption - desorption isotherm and pore size distribution of TiO_2 and SDBS/C/Nd/TiO2; e) - f) XPS spectra of TiO2 and SDBS/C/Nd/TiO_2

3.3. UV-vis-NIR diffuse reflectance spectroscopy

As can be seen from Figure 2b, absorption in the wavelength range less than 400 nm is obviously determined by the inherent absorption characteristics of TiO_2 and the absorption edge of SDBS/C/Nd/TiO_2 catalyst moves towards the long wave direction. Therefore, the band gap of TiO_2 and SDBS/C/Nd/TiO_2 is 3.1eV and 2.78eV, respectively (as shown in Table 1). The absorption performance of SDBS/C/Nd/TiO_2 in the ultraviolet region is enhanced compared with pure TiO_2. This shows that the adding of anionic surfactant and the doping of elements change the absorption performance of TiO_2, which makes the catalyst SDBS/C/Nd/TiO_2 have higher absorption in the ultraviolet region. Two absorption peaks appear at the wavelength of 550 and 590 nm, which is a unique property of Nd element, thus demonstrating that Nd^{3+} ions are successfully doped into the catalyst. Because the doped element changes its internal energy band structure

and electronic structure, intermediate energy levels may be generated between the valence band and conduction band of TiO_2, and photogenerated electrons and holes can transition through these intermediate energy levels. The required excitation energy is reduced to the visible light range, so that doped TiO_2 has visible light absorption and absorption energy. Thus, it is beneficial to improve the utilization rate of visible light.

3.4. N_2 adsorption-desorption

Figure 2c shows that the relative pressure range is 0.8-1, and both catalysts have a hysteresis ring, indicating that both catalysts have pore structures. The specific surface area, pore volume and pore diameter of different catalytic materials can be obtained by BET model and BJH model. According to the classification of BDDT, the N_2 adsorption-desorption isotherm of the prepared catalytic material belongs to type IV. It can be seen from Figure 2d the pore size distribution curve that the pore size of TiO_2 and SDBS/C/Nd/TiO_2 catalytic materials is concentrated between 30 nm and 50 nm, which belongs to mesoporous materials. The specific surface area and pore volume of TiO_2 and SDBS/C/Nd/TiO_2 are 55.22 m^2g^{-1}, 0.089 cm^3g^{-1} and 116.1 m^2g^{-1}, 0.361 cm^3g^{-1}, respectively. The results are shown in Table 1. According to the data in Table 1, compared with pure TiO_2, the pore volume of SDBS/C/Nd/TiO_2 is smaller than that of pure TiO_2, and the corresponding specific surface area is larger than that of pure TiO_2, which indicates that the specific surface area of the catalytic material can be improved to a certain extent after adding anionic surfactants and doping with elements. Such a large specific surface area and porous structure for SDBS/C/Nd/TiO_2 are conducive to the rapid diffusion of reactants and products, thus promoting the improvement of photocatalytic activity. The large pore volume provides a good mass transfer space for dynamic reactants and reduces steric hindrance. It helps to speed up the reaction rate. Thus, SDBS/C/Nd/TiO_2 catalytic material has higher photocatalytic activity.

3.5. X-ray photoelectron spectroscopy

Figure 2e-h shows the full spectrum of SDBS/C/Nd/TiO_2, C 1*s* spectrum, Nd 3*d* spectrum and Ti 2*p* binding energy change spectrum, respectively. As can be seen from Figure 2e, both C and Nd elements have been successfully doped into TiO_2. Combined with XPS data analysis, the characteristic peaks of C 1*s*, Nd $3d^3$, Nd $3d^5$, Ti 2*p* and O 1*s* are located around 288 eV, 1002 eV, 982 eV, 458.78 eV and 530 eV. Comparing with the Ti 2*p* binding energy of TiO_2 and SDBS/C/Nd/TiO_2 (Figure 2h), the orbital electron binding energy of Ti 2*p* of SDBS/C/Nd/TiO_2 moves to the low energy region. This is due to the fact that Nd is less electronegative than Ti, which causes the electron cloud around Ti to become denser.

3.6. Catalytic activity

The photocatalytic activity of the catalysts in visible light was evaluated according to the decolorization rate of methyl orange solution. The results are shown in Figure 3. From Figure 3a, catalysts under the condition of visible light has a certain degradation ability, the SDBS/C/Nd/TiO_2 have larger decolorization rate, which can achieve 67%, mainly because SDBS/C/Nd/TiO_2 has larger specific surface area and pore volume, thus the larger mass transfer space is provided for photocatalytic and it is favorable for the improvement of photocatalytic activity.

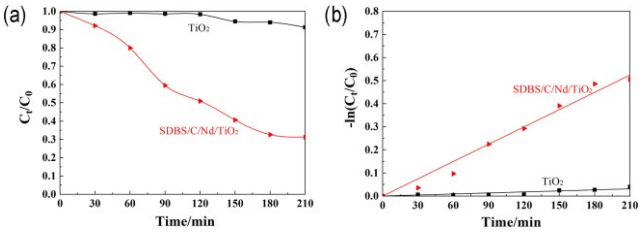


Figure 3. a) The degradation of methyl orange dye by catalytic materials; b) the first order kinetic response spectra

The decolorization rate of methyl orange solution follows the first-order kinetic model, as shown in Figure.3b, which can be expressed as -ln (C_t/C_0) = K*t*, Where, C_0 is the initial concentration of methyl orange solution; C_t is the concentration of light t min methyl orange solution; K is the rate constant; higher K means higher photocatalytic activity. The photocatalytic rate constant of SDBS/C/Nd/TiO_2 is higher than that of TiO_2 catalyst, which indicates that the photocatalytic activity of SDBS/C/Nd/TiO_2 is significantly higher than that of TiO_2 catalyst.

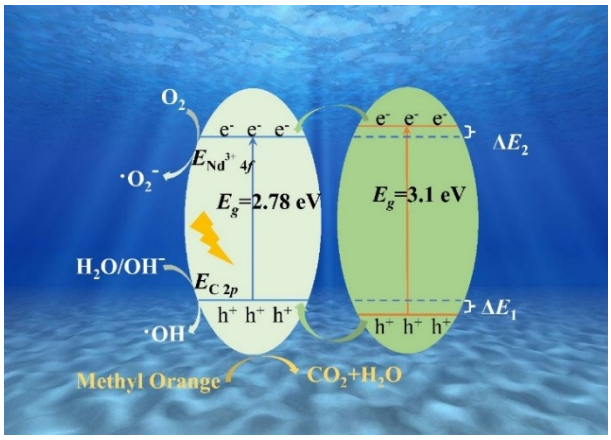


Figure 4. The photocatalytic possibility mechanism of SDBS/C/Nd/TiO2

3.7. Study on photocatalytic activity mechanism

According to the experimental characterization of SDBS/C/Nd/TiO_2, the possibility mechanism of photocatalysis of SDBS/C/Nd/TiO_2 catalyst is inferred as shown in Figure 4. The synergistic effect of non-metallic C and rare earth metal Nd makes the band gap of SDBS/C/Nd/TiO_2 narrower. The doping of C element with 2*p* empty orbital into TiO_2 can produce a new energy band $E_{C\ 2p}$, which is above the valence band (VB) of TiO_2. When Nd is doped in TiO_2, a new energy band $E_{Nd^{3+}\ 4f}$, which is lower than TiO_2 conduction band (CB), is generated due to the empty orbital of Nd 4*f*, forming the energy difference ΔE_1 and ΔE_2. Thus, the photocatalytic performance of SDBS/C/Nd/TiO_2 catalyst is improved. At the same time, O_2 and H_2O/OH^- adsorbed on the surface of nanoparticles capture electrons and holes, and undergo REDOX reaction to produce $\bullet O_2^-$ and $\bullet OH$, oxidizing organic pollutants to CO_2 and H_2O, reduces the recombination of

photogenerated electrons and holes, and is more conducive to enhancing photocatalytic activity. Sodium dodecyl sulfate, as a surfactant, improved the surface morphology of TiO_2 by sol-gel process and found that TiO_2 modified by non-metallic C and rare earth metal Nd showed higher photocatalytic efficiency. SDBS/C/Nd/TiO_2 photocatalyst has a wide range of applications in the environmental field, such as air purification, water treatment, and can also decompose harmful gases in the air, effectively removing pollutants in indoor air.

4. Conclusion

In summary, SDBS/C/Nd/TiO_2 and pure TiO_2 catalysts were prepared by sol-gel method. According to experimental results, the morphology and crystal form of the SDBS/C/Nd/TiO_2 catalyst were not significantly changed, but the absorption performance of the catalyst was improved, and the specific surface area was increased after the doping of C and Nd. After using SDBS as the template agent, the surface particle dispersion of SDBS/C/Nd/TiO_2 is improved and the agglomeration phenomenon is reduced. The crystal type of the catalyst is still anatase after doping, and the absorption edge of light has a certain degree of red shift, which is conducive to enhancing the absorption of visible light. Based on photocatalytic experiment, the SDBS/C/Nd/TiO_2 degradation of methyl orange solution is higher than pure TiO_2, the decolorization rate can reach 67%, mainly because not only new impurity levels is generated at the top of the valence band, but also the position of the bottom of the conduction band decreases more, making the band gap of the SDBS/C/Nd/TiO_2 narrower, which was conducive to widening the photoabsorption range of the catalyst, thus improving the photocatalytic performance of the SDBS/C/Nd/TiO_2 catalyst.

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