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The Physical and Photocatalytic Properties of N-doped TiO_2 Polycrystalline Synthesized by a Single Step Sonochemical Method at Room Temperature

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Abstract. Titanium dioxide (TiO_2) is one of popular semiconductor materials that usually used for photocatalytic application. Recent studies show the improvement of TiO_2 photocatalytic activity through nitrogen doping (N-doped TiO_2). In this study, we focused on the synthesis and characterization of N-doped TiO_2. Ultrasonic assisted synthesis or sonochemical method was used to prepare N-doped TiO_2 polycrystalline powder under room temperature. X-ray diffractometer (XRD), and diffuse reflectance ultraviolet-visible spectrophotometer (DR-UV) were employed to evaluate physical properties of N-doped TiO_2. XRD pattern exhibited that all samples have anatase crystalline phase and crystallite size decrease with increase of N dopant concentration. The absorbance spectra showed the slight shift toward higher wavelength (red shift) and from Kubelka-Munk function the band gaps were getting smaller with increase of N content. The increase of photocatalytic activity under solar radiation was achieved by N-doped TiO_2 samples with highest efficiency about 81 % for 5% of N doping concentration.

Introduction

TiO_2 is known as the most observed semiconductor materials for many applications, such as energy and environment. Many studies show that TiO_2 has highly effective material for photocatalyst [1], solar cells [2,3], gas sensors [4], lithium batteries [5], self-cleaning materials [6] and so on. Since Fujishima and Honda reported the first time the photoelectrochemical water splitting into H_2 and O_2 [7], many researchers have been investigating TiO_2 to create clean hydrogen production from water and solar energy. TiO_2 has attracted increasing attention because of its chemical stability, low cost, nontoxicity and ease of availability [8]. However, the bandgap of TiO_2 is high about 3.2 eV which needs the high energy for activation such as ultraviolet (UV) light. In solar spectrum, only 5 % spectrum of UV light that can be utilizedfor photo-activation. The highest spectrum is visible light about 45 %,but its energy is lower than 3 eV so that difficult to activate TiO_2. Many efforts have been done to create an efficient visible light activated TiO_2 photocatalyst such as substitution doping of metal and non-metal ions, ion implantation, organic dye sensitization and hydroxide or surface coordination [9]. Many studies reported that metal and non-metal doping contribute to increase photocatalytic activity under visible light irradiation.Instabilities is one of a problem caused by metal doped materials [10]. Non-metal doping is more favorable because of its low-cost and economically friendly [11]. Nitrogen is promising element that can be used to enhance the photocatalytic activity of TiO_2 under visible light irradiation due to its comparable atomic size with oxygen, metastable center formation, small ionization energy and stability [12]. There are many methods to prepare N-doped TiO_2 powders such as heat treatment [13], sol-gel [14,15], mechanochemical technique [16], solvothermal [17], and sonochemical technique [11,12]. Many studies showed that sonochemical method can be used to obtain many novel materials with unusual

properties [18-21]. The ultrasound will affect the chemical reaction because of acoustics cavitation within liquid known as the formation, growth and explosive collapse of bubbles. This effect produces strange chemical and physical condition. The interval of chemical transformation will be very fast under this condition with temperatures of around 1000 °C, pressures of about 1000 atm, and heating and cooling rates above 10 billion °C/second [22]. In this study, we prepared N-doped TiO_2 powder by sonochemical method under room temperature. The obtained powders were characterized by XRD and DR-UV to analyze the structural and optical properties, respectively. Methylene blue (MB) was used as organic pollutant to test the photocatalytic activity of N-doped TiO_2 under solar radiation.

Experimental Method

Titanium tetraisopropoxide or TTIP, ($Ti[(CH_3)_2CHO]_4$) from Aldrich was used as TiO_2 source. TTIP (5 mL), acetone (1 mL) and methanol (1mL) were mixed under room temperature by magnetic stirrer. Urea from Merck was added into the each solution as nitrogen (N) source with concentration 0%, 1%, 3%, 5%, 7%, and 9% at. and we simply called the samples as TiO_2, NT1, NT2, NT3, NT4 and NT5. All chemicals were used without any further purification. Then, the each solution was irradiated by ultrasonic with frequency about 40 kHz for 40 minutes at room temperature. The solvent was evaporated by hotplate magnetic stirrer under temperature about 60 °C untill dry and the white powder was obtained. Furthermore, the powder was heated at 500 °C for 2 hours and a pale yellow of N-doped TiO_2 powder was obtained. Shimadzu Maxima 7000 XRD was used to characterize the structural property of prepared TiO_2 with Cu-Kα wavelength about1.5405 Å. The optical characteristics was evaluated by Shimadzu 2450 DR-UV spectrophotometer. In order to evaluate the photocatalytic activity, N-doped TiO_2 (0.02 g) was immersed into MB, (50 mL) under solar radiation. The concentration of MB was 20 mg/L with water as solvent. The absorbance of MB solution was measured by Shimadzu1240 SA UV-Vis spectrophotometer every 10 minutes.

Results and Discussions

XRD analysis

Figure 1 shows the XRD patterns of the obtained TiO_2 and N-doped TiO_2 powders by sonochemical method under room temperature. The incident angle was adjusted at 0.02° and 2θ was scanned in the range of 10-90°. The X-ray diffraction peaks exhibit that all samples have anatase TiO_2 crystalline phase (JCPDS 21-1272) and there is no peak for brookite and rutile phase was detected. This result indicates that anatase TiO_2 powder can be obtained by a single step sonochemical method. We also see that no nitrogen-derived peaks can be detected in the all samples. The amounts of nitrogen may be less than detection limit of XRD or the element is difficut to be identified in TiO_2 framework [23]. The peaks (2θ) at 25.44°, 38.02°, 48.02°, 54.14°, and 55.14° represent (101), (112), (200), (105) and (211), respectively. Table 1 shows the crystallite size of N-doped TiO_2 which was calculated from the broadening of (101) XRD peak by the following Scherrer's equation.

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

Where D is the crystallite size, K is the Scherrer's factor, λ is the X-ray wavelength of CuK_α (0.154nm), β is the full-width at half maximum (FWHM) and θ is the Bragg angle.

The crystallite size of samples decrease with addition of nitrogen. It caused by interchange of Ti atoms (atomic radius of 76 pm) with N atoms (atomic radius of 56 pm). Kim and co-workers found that incorporation of N restrict the growth of TiO_2 crystallites [23]. We also can see the FWHM of N-doped TiO_2 higher than pure TiO_2. It indicates that the crystallinity of sample reduce with addition of N. Some defects may be occurred in the crystal structure of TiO_2. This defects also may affect the slight shift of 2θ angle forthe (101) plane.

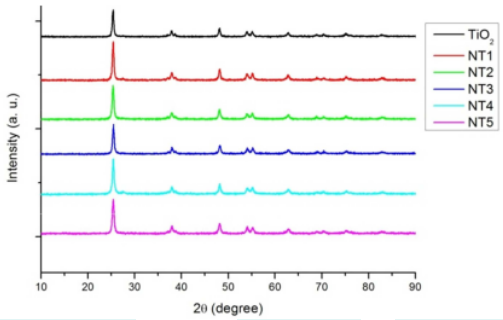


Figure 1. XRD analyses of TiO_2 and N-doped TiO_2.

Table 1. Peak angle (2θ), FWHM and crystallite size of samples at (101).

Sample	2θ (°)	FWHM (rad)	D (nm)	Eg (eV)
TiO_2	25.42	0.00728	19.26	3.26
NT1	25.42	0.00762	18.39	3.15
NT2	25.36	0.00848	16.45	3.42
NT3	25.48	0.00814	17.28	3.20
NT4	25.42	0.00846	16.57	3.04
NT5	25.44	0.00879	15.96	2.90

Optical properties

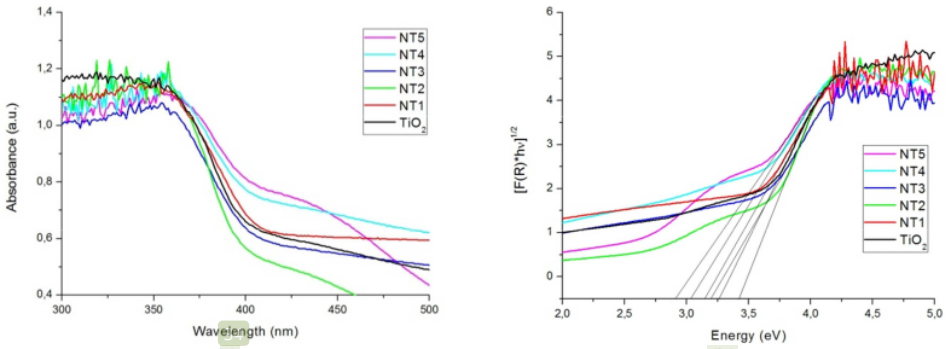
The absorbance spectra of as-synthesized samples were shown in Figure 2(a). It can be seen that almost all the doped TiO_2 samples slightly shifted to higher wavelength, exception for NT2. Yang and co-workers state that the red-shift of N-doped TiO_2 absorbance spectra is due to incorporation of N in crystalline lattice of TiO_2 [24]. Kubelka-Munk function (eq. (2) and (3)) was used to estimate the optical band gap of as prepared samples from reflectance data.

$$F(R_\infty) = (1-R_\infty)^2 / 2R_\infty \quad (2)$$

Where $F(R_\infty)$ is equivalent to absorption coefficient and R_∞ is reflectance [25-28]. For an indirect semiconductor, the absorption coefficient can be expressed as,

$$[F(R_\infty) * hv]^{\frac{1}{2}} = A(hv - Eg) \quad (3)$$

Where h is Planck constant, v is frequency of light, A is constant and Eg is the band gap. Therefore, Kubelka-Munk function can be constructed by plotting $[F(R_\infty) * hv]^{0.5}$ versus energy (Fig 2(b)), if $[F(R_\infty) * hv]^{0.5} = 0$ we will get the band gap and the results were shown in Table 1. The band gaps were obviously narrower than pure TiO_2 which was important to improve the photocatalytic properties [15]. The decrease of band gap might be caused by the formation of oxynitride and the substitution of the lattice oxygen by nitrogen [29,30].



(a) (b)

Figure 2. (a) Absorption spectra of all prepared samples, and (b) Diffuse reflectance UV-vis spectra of all prepared samples.

Photocatalytic activity

MB was used as pollutant for photocatalytic evaluation of as-synthesized samples under solar radiation, the result was shown at Figure 3. Without photocatalyst (photolysis), MB was highly difficult to degrade. TiO_2 particles achieved about 53% degradation efficiency. Compared to TiO_2, it was obvious that the N-doped samples were more effective with the highest degradation efficiency about 81% for NT5 sample.This photocatalytic improvement might be caused by the increase of photoabsorption in the visible light region. Jia and co-workers stated that the incorporation of nitrogen ions increase the interfacial charge transfer and prevent from charge recombination [31]. TiO_2 photocatalyst produce hydroxyl ion and superoxide that effectively destroy organic pollutant [32].

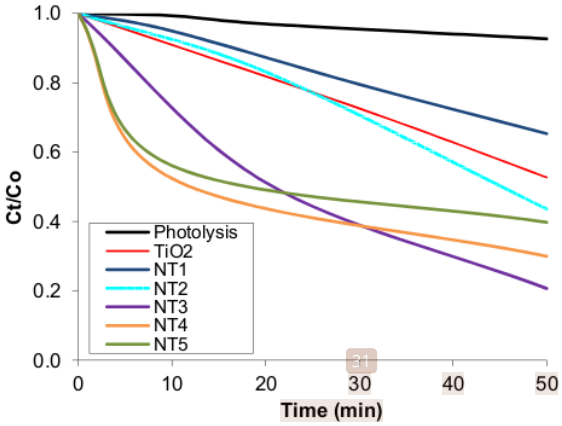


Figure 3. Photocatalytic degradation of MB by as-prepared samples under solar radiation.

Conclusion

N-doped TiO_2 particles have been successfully synthesized by a single step sonochemical method at room temperature. XRD pattern showed that all samples have anatase crystalline phase. Addition of N reduced the crystallite size and slightly shifted the diffraction angle of (101) plane. The band edge of absorbance spectra shifted to higher wavelength and from Kubelka-Munk function, the band gap of N-doped samples were getting smaller compared to pure TiO_2 sample. The photocatalytic efficiency increase about 81% for N-doped $TiO_2$5% sample.

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