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Simple and Cost-Effective Precipitation Synthesis and Characterization of Bi-doped TiO₂ Nanocomposite

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ABSTRACT

In this study, Bi/TiO₂ was successfully synthesized by simple and cost-effective precipitation method. Titania has drawn increasing attention among diverse oxide photocatalysts due to its biological and chemical inertness, strong photo oxidization power, cost effectiveness, and long-term stability against light and chemical corrosion. The composite Bi/TiO₂ was characterized by means of scanning electron microscopy (SEM) with elementary dispersive X-ray analysis (EDX) and X-ray diffraction (XRD) analysis. It's crucial and difficult to find anatase TiO₂ with high thermal stability and photocatalytic activity. Heat treatment, on the other hand, affects photocatalytic activity by changing the textural properties, crystallinity degree, and surface chemical states. The great thermal stability of the as-prepared TiO₂ nanosheets is due to high crystallization (low impurity) and oxygen vacancy. Surprisingly, high photocatalytic activity for photocatalytic oxidation breakdown of acetone in air under UV light illumination.

Key words: Nanoparticles, Composite, Semiconductor, Titanium dioxide

Semiconductor metal oxides such as titanium dioxide (TiO₂) and zinc oxide (ZnO) have attracted much attention in recent years due to their various applications to the photocatalytic degradation of organic pollutants in water, dye sensitized photovoltaic solar cells and inorganic antibacterial agents [1-5]. TiO₂ in anatase form and ZnO in wurtzite phase are the most used photocatalysts due to their electronic band structure. The band gap energy (3.2 eV) and the conduction band edge position of these semiconductors are very similar ($E_{CB} = -0.52$ V at pH 5–7 Vs NHE). Both are efficient photocatalysts [6-8]. Oxide semiconductor photocatalysts with high activity for environmental protection operations such as air purification, water disinfection, hazardous waste cleanup, and water purification have received a lot of attention in recent years [1–5]. Titania has drawn increasing attention among diverse oxide semiconductor photocatalysts due to its biological and chemical inertness, strong photo oxidisation power, cost effectiveness, and long-term stability against light and chemical corrosion [6–13].

Due to its commercial usage in high-temperature settings, photo-active anatase TiO₂ with higher thermal stability is now required [8-10]. However, under normal conditions, the highly photoactive anatase phase is irreversibly changed to the less reactive rutile phase at around 600 °C, limiting its

usefulness for high-temperature applications [11,12]. As a result, increasing the anatase structure's stability temperature and maintaining strong photocatalytic activity at high temperatures is critical [13]. Doping has been used extensively to improve the thermal stability of anatase titania. Non-metal ions dopants including S, N, and F [14-17] have been utilised to raise the temperature of the anatase-to-rutile phase transition. Nano-TiO₂ is preferred in anatase nature because of its high photocatalytic performance, since it has high reactivity, high specific area, nontoxicity and low cost [9]. Doping is an easy strategy for enhancing the catalytic behavior of the nanocomposites. Doping of TiO₂ with metal ions (nickel, iron, and vanadium) or nonmetallic (sulfur, nitrogen and carbon) [10-16]. Among these doping, transition metal doped TiO₂ was effective method [17].

MATERIALS AND METHODS

Bismuth nitrate pentahydrate (Bi(NO₃)₃·5H₂O), Titanium tetraisopropoxide (Ti(iso-OC₃H₇)₄), ethanol (C₂H₅OH) and hydrochloric acid (HCl) were the guaranteed reagents of Sigma Aldrich and used as such.

Synthesis of Bi-doped TiO₂ nanocomposite

Bi-doped TiO₂ was synthesized by precipitation method. Ethanol (40 mL) was added to titanium tetraisopropoxide (20mL), to which an aqueous ethanolic solution (20 mL water + 40 mL ethanol) was added drop wise. This resulted in the formation of a white precipitate (solution A). A calculated amount of Bi(NO₃)₃·5H₂O was added to appropriate amounts of ethanol, double distilled water and HCl making a total of 40

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mL of reaction mixture volume (solution B). It was added to the solution A followed by stirring for 6 h and ultrasonic dispersion for 40 minutes. The obtained materials were filtered, washed and dried overnight at 100°C then ground to obtain a powder. It was further calcination treatment at 550°C for 1 h.

Characterization methods

Scanning electron microscopy and elementary dispersive X-ray analysis experiments were carried out on a FEI Quanta FEG 200 instrument with EDX analyzer facility at 25°C. The sample was prepared by placing a small quantity of prepared nanocomposites on a carbon coated copper grid and allowing the solvent to evaporate. X-ray diffraction spectra was recorded on the X'PERT PRO model X-ray diffractometer from Pan

Analytical instruments operated at a voltage of 40 kV and a current of 30 mA with Cu K α radiation.

RESULTS AND DISCUSSION

SEM with EDX analysis

The SEM image of the Bi/TiO₂ is shown in (Fig 1a). From the SEM images of the nanocomposite possess a spherical shape structure with the size range of ~20 nm. EDX analysis confirms Bi, Ti and O are present in composite material (Fig 1b), which provides direct evidence that the Bi particles were entrapped with in TiO₂ lattice.

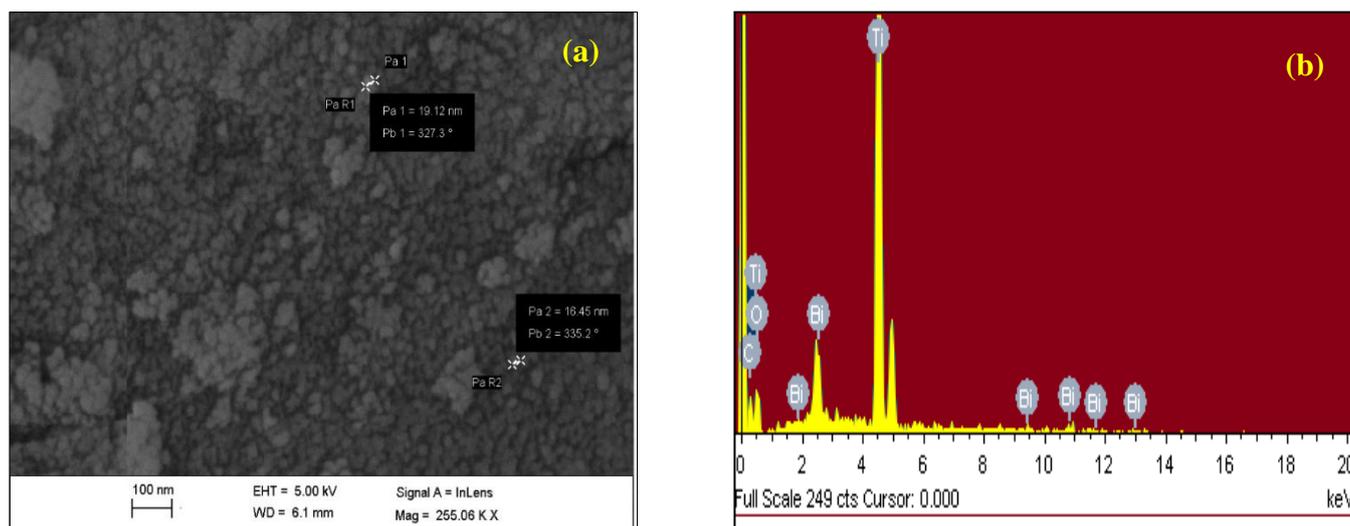


Fig 1(a) SEM images of Bi/TiO₂ and (b) EDX analysis of Bi/TiO₂

XRD analysis

The obtained XRD of the Bi/TiO₂ was shown in (Fig 2). The peaks at 22.4°, 25.4°, 40.8°, 44.5°, 47.5°, 54.4° and 69.5° are the diffractions of the Bi (003), TiO₂ (101), Bi (110), (015) and TiO₂ (200), (105), (116) crystal planes, respectively (JCPDS no. 89-4921, 85-1330 and 21-1272). It has been reported that the doping of Bi³⁺ into TiO₂ lattice.

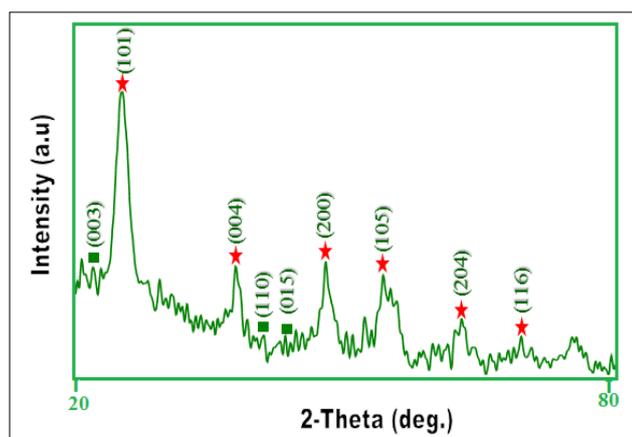


Fig 2 XRD spectra of Bi/TiO₂

Photocatalytic activity

The photocatalytic degradation of acetone in air was used to assess the photocatalytic activity of TiO₂ samples calcined at various temperatures. With increasing irradiation time, the concentration of acetone falls and the quantity of emitted CO₂ increases during the photocatalytic reaction. It can be noticed that the carbon dioxide products to acetone destroyed

ratio was close to 3:1. Under the same conditions, the photocatalytic activity of Degussa P-25 TiO₂ (P25), which is well known for having good photocatalytic activity [20], was also investigated. T200 has a rate constant of $12.5 \times 10^{-3} \text{ min}^{-1}$, which is 2.1 times that of P25 ($5.94 \times 10^{-3} \text{ min}^{-1}$). This is due to the surface fluorination, greater BET surface area ($62.4 \text{ m}^2/\text{g}$), and exposed reactive 001 facets of T200. Surface fluorination has been shown to significantly improve TiO₂ photocatalytic activity by forming free $\cdot\text{OH}$ radicals. The crystalline phase, crystallinity, particle size, surface area, and surface chemical state of TiO₂ all affect the degradation efficiency of organic contaminants. However, these characteristics frequently conflict with one another, affecting TiO₂'s photocatalytic activity. For example, because a significant amount of adsorbed organic molecules promotes the photocatalytic process, a large specific surface area may be an important factor influencing the rate of photocatalytic degradation reaction. However, large specific surface area powders are frequently associated with substantial amounts of crystalline flaws or weak crystallization, which promote photo-generated electron and hole recombination, resulting in low photocatalytic activity.

The photocatalytic activity of TiO₂ is clearly reduced when the calcination temperature is increased to 600 °C. T600's rate constant drops to $2.19 \times 10^{-3} \text{ min}^{-1}$, which is barely 18% that of T200. This can be explained by surface fluorine desorption (and a decrease in BET surface areas. T600 nanosheets have a BET surface area of $15.6 \text{ m}^2/\text{g}$, which is less than a fifth of that of T200 nanosheets ($62.4 \text{ m}^2/\text{g}$). However, following calcining at 700 °C ($4.79 \times 10^{-3} \text{ min}^{-1}$ for T700), the photocatalytic activity of TiO₂ begins to rise, which is attributed to increased crystallization. The photocatalytic activity of TiO₂ rapidly diminishes as the calcination temperature rises, due to a

decrease in BET surface areas and the percentage of exposed high-energy [001] facets. It's interesting to note that after being calcined at 1100 °C, the photocatalytic activity of the T1100 sample ($3.44 \times 10^{-3} \text{ min}^{-1}$) is still roughly 60% that of the P25 sample. The photocatalytic activity of TiO_2 powders significantly drops ($0.68 \times 10^{-3} \text{ min}^{-1}$) when the calcined temperature exceeds 1200 °C due to severe sintering of the samples and the production of rutile phase [44].

CONCLUSION

Bi/TiO_2 has been synthesized successfully by a simple and cost-effective precipitation method. The presence of Bi into TiO_2 lattice has been revealed by XRD and SEM with EDX analysis. Results confirmed the formation of Bi/TiO_2 nanocomposite. The SEM and XRD analysis showed the Bi/TiO_2 has average particle size of 20 nm has been confirmed.

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