Abstract

Titanium dioxide (TiO₂) is a semiconductor material that plays an important role in photocatalysis. Bicontinuous concentric lamellar (bcl) is an interesting morphology with an open channel pore structure that has been successfully synthesized on silica-based materials. If bcl morphology can be applied in TiO₂ system, then many surface properties of TiO₂ can be enhanced, i.e. photocatalytic activity. A simple and effective strategy has been demonstrated to transform aggregated and spherical TiO₂ particles to bcl morphology via alkaline hydrothermal route. Alkaline hydrothermal treatment successfully transforms TiO₂ particle surface to have bcl morphology through swelling with ammonia then followed by phase segregation process. We proposed this strategy as a general pathway to transform the particle surface with any shape to have bcl morphology. Copyright © 2019 BCREC Group. All rights reserved

Keywords: Alkaline hydrothermal treatment; bcl morphology; lamellar morphology; modified morphology; nanostructured TiO₂

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1. Introduction

Titanium dioxide (TiO₂) is an important inorganic semiconductor material in various applications due to its photosensitive, photostable, and environmentally friendly properties [1]. The advantages properties of TiO₂ provide potential applications in many fields such as photocatalysts [2], dye-sensitized solar cells [3], and catalyst support [4]. Due to its wide application, research about synthesis, characterization and fundamental understanding of TiO₂ material has been intensively studied in last recent years. Many researchers developed TiO₂ synthesis methods to enhance the TiO₂ activity in various applications [5,6], especially as photocatalysts.

Many research groups reported that particle size and morphology are important to obtain TiO₂ with good photocatalytic activity [7-9]. The effective charge transfer in photocatalyst TiO₂ requires the presence of surface active sites [10].
Have a high surface area is the essential requirement to have high surface-active sites photocatalyst. The common strategy to increase surface area is by reducing the crystallite size. However, small TiO$_2$ particles have low mechanical strength and easily agglomerate to form larger particles. We can use another strategy to increase the surface area of TiO$_2$ particles by modifying its morphology. Conventional mesoporous TiO$_2$ has only closed channel pore structure from intercrystallite spaces. However, closed channel pore structure has several drawbacks, such low accessibility due to diffusion limitation, pore blocking, and difficult pore surface activation, despite its high selectivity. Therefore, in vast reaction schemes, the open channel is more favorable. TiO$_2$ particles with open channel structure provide high surface area, high accessibility and facile pore surface modification [11]. Photocatalytic activity of mesoporous materials with open channel pore structure is easy to adjust chemically due to large reachable surfaces [12].

One of morphology with an open channel pore structure that attracts much attention is bicontinuous concentric lamellar (bcl) morphology. bcl morphology has been successfully synthesized in various type silica-based materials using reverse micelle templating method [13]. The morphology of bcl silica is quite unique. Here, uniform spherical particles consist of bicontinuous lamellae arranged concentrically providing high surface area, large pore volume, and high accessibility [13]. If this morphology can be applied in TiO$_2$ system, then many surface properties of TiO$_2$ can be enhanced, i.e. photocatalytic activity. Synthesis of TiO$_2$ with reverse micelle has been reported elsewhere, but only TiO$_2$ particles with irregular morphology obtain in the form of aggregates. This type of particles provides low photocatalytic activity [14]. bcl morphology cannot be obtained using the reverse micelle templating due to the hydrolysis nature of TiO$_2$ precursors, i.e. TiCl$_4$, TTIP, etc. It is a rule of thumb that TiO$_2$ precursors hydrolysis rapidly event in the traces of water.

Herein, we demonstrate the transformation of aggregated and spherical TiO$_2$ particles to bcl morphology via alkaline hydrothermal treatment. Synthesis of nanostructured TiO$_2$ with bcl morphology will be carried out in two simple steps without surfactant. Slow hydrolysis is the main strategy to form TiO$_2$ particles with controllable particle shape. Swelling and phase segregation is the key to obtain bicontinuous lamellar morphology.

2. Materials and Methods

2.1 Preparation of Nanostructured TiO$_2$ with bcl Morphology

Titanium dioxide was synthesized by modifying previously reported method [5]. First, 1.1 mL of Titanium (IV) isopropoxide (TTIP, 99%, Sigma--Aldrich) was added to 100 mL of ethanol (99%, Merck) under vigorous stirring for 30 minutes until milky white suspension formed. It was kept static for 12 hours and then filtered. The powder obtained was washed with deionized (DI) water and ethanol, and dried at 60 °C for 6 hours. Then, 0.3 g of as-prepared TiO$_2$ was dissolved in 24 mL of ammonia (25%, Sigma--Aldrich) together with 12 mL of DI water. The mixture was transferred to a 50 mL Teflon-line stainless steel autoclave and heated to 120 °C for 6 hours. After that, the autoclave was cooled down to room temperature naturally. The obtained products were filtered and washed with DI water and ethanol. Finally, the powder obtained was calcined at 500 °C for 1 hour. All the steps above are repeated using the same method with absolute ethanol is replaced by DI water.

Within this study, the samples are designated as TiO$_2$-A (aggregated as-synthesized), TiO$_2$-S (spherical as-synthesized), TiO$_2$-A-HT (aggregated after hydrothermal treatment), TiO$_2$-S-HT (spherical after hydrothermal treatment), TiO$_2$-A-HT-500 (aggregated after hydrothermal treatment and calcination at 500 °C), and TiO$_2$-S-HT-500 (spherical after hydrothermal treatment and calcination at 500 °C).

2.2 Sample Characterizations

The morphology of obtained samples was characterized with scanning electron microscopy (SEM, Hitachi SU3500) with the accelerating voltage at 10 kV. The phase structures were determined by Raman spectroscopy (Bruker – Senterra) with a 532 nm Nd:YAG DPSS (diode-pumped solid-state) laser at 2 mW on 5s scanning time and X-ray diffraction (XRD, Bruker D8 Advance with LynxEye XE detector) on a Scintag diffractometer with CuKα radiation ($\lambda = 1.54060$ Å) at a scanning rate of 0.239° s$^{-1}$ in the 2θ range from 10 to 90°. Photoluminescence signals were measured by a UV-visible fiber spectrometer (USB4000 Ocean optics) coupled to a 120x magnification microscope objective lens equipped with a 405 nm long-wave pass edge filter (Semrock Edge-Basic, BLP01-405R-25). The sample was excited by a 405 nm diode laser equipped with a 405 nm laser clean-up filter (Semrock MaxLine,
3. Results and Discussion

Nanostructured TiO$_2$ with bcl morphology was prepared as explained in the Experimental Section. As shown in Figure 1a and 1b, the aggregate and spherical shape particles were investigated by SEM. The main difference in preparation condition between two kinds of resulted particles is the hydrolysis rate of TiO$_2$ precursor. TTIP dissolved in DI water experiences rapid hydrolysis, thus produces aggregated particles with an irregular shape. On the other hand, if DI water was replaced by ethanol, TTIP experiences a slow hydrolysis pathway to producing spherical particles.

Figure 1c and 1d show the morphology of particles after alkaline hydrothermal treatment. We observed the surface of both aggregates and spherical particles transform into bi-continuous lamellar morphology. Alkaline hydrothermal treatment has been proved to be independent of particle shape and synthesis history. If the particles have not been calcined yet, then the method can be applied. Based on results above, we hypothesized that the formation of these lamellar structures was carried out by chemically modifying the surface of particles through swelling and phase segregation under alkaline condition. Figure 1e and 1f show the SEM images of samples after calcination at 500 °C for 6 h. Both images show no significant changes in their morphology after calcination process with respect to Figure 1c and 1d, respectively. Although SEM images do not show noticeable morphology changes after the calcination process, Raman, XRD, and BET analysis reveal significant differences.

Figure 2a shows Raman spectra of synthesized TiO$_2$ samples. In principle, Raman analysis is not a sensitive method to distinguish particle morphology, i.e., both uncalcined aggregated and spherical particles give relatively similar Raman spectra. However, Raman is very sensitive to distinguish different chemical components and polymorphism in crystalline materials, i.e., between anatase and rutile TiO$_2$ polymorphs. In sample TiO$_2$-A and TiO$_2$-S shows broad bands at 200, 450, and 610 cm$^{-1}$. These peaks were frequently observed in amorphous TiO$_2$ [15]. In sample TiO$_2$-A-HT and TiO$_2$-S-HT, the peaks are seen at 190, 270, 440, and 650 cm$^{-1}$. These peaks due to the formation of titanate species during alkaline hydrothermal process [15]. Moreover, aggregated particles (TiO$_2$-A-HT) provide spectra with richer features and sharper peaks with respect to spherical particles (TiO$_2$-S-HT). This might due the aggregated particles have more defects on their surfaces, which is in good agreement with SEM results depicted in Figure 1c and 1d, respectively.

After calcined samples demonstrate typical anatase peaks with Raman shift at 154.50,
206.04, 396.18, 513.34, and 631.73 cm\(^{-1}\) that assigned to the vibrational modes \(E_g(1), E_g(2), B_{1g}(1), B_{1g}(2)+A_{1g}\), and \(E_g(3)\), respectively \[16\]. These peaks represent the vibrational normal modes of anatase as depicted schematically in Figure 2b. Rutile peaks cannot be observed in a calcined sample without deconvolution. In other hands, there are no anatase or rutile peaks can be observed in Raman spectra of uncalcined samples.

To get more structural insights of synthesized TiO\(_2\), XRD analysis must be performed. Figure 3 shows the diffractogram of all samples, including the reference standard of rutile and anatase polymorphs. The inset of Figure 3 shows the crystal structure of anatase and rutile. Uncalcined samples (TiO\(_2\)-A, TiO\(_2\)-S, TiO\(_2\)-A-HT, and TiO\(_2\)-S-HT) demonstrate similar XRD pattern consists of three broad amorphous peaks at 15.3-35.6°, 42.4-52.3°, and 57.2-70.1° \[18-20\]. The calcined samples show dominant anatase phase with a trace rutile phase. The peaks at 25.3, 36.9, 37.8, 38.7, 48.0, 53.9, 55.1, 62.1, 62.7, 68.7, 70.3, 75.0, 76.1, and 82.7° are assigned to the anatase phase \[21\]. A trace peak at 27.48° is assigned to the rutile phase \[22\].

Based on XRD and Raman analysis results, we propose that the structural precursor of anatase phase obtained after calcination has already resided in all uncalcined samples. The structural precursor feature is more pronounced in XRD than in Raman results, represents by three broad amorphous peaks. Structural precursor appears in Raman spectra only as a broad background. Alkaline hydrothermal treatment does not alter much this feature in XRD pattern, only one additional peak appears around 48.05°, represents the anatase phase. This structural precursor feature can be used to indicate where the calcination process will terminate. In this case, the calcination of this material mainly leads to the formation of the anatase phase.

As shown by Raman and XRD measurements, both of TiO\(_2\)-A-HT and TiO\(_2\)-S-HT are in amorphous state and turn into anatase crystalline phase after calcination at 500 °C (TiO\(_2\)-A-HT-500 and TiO\(_2\)-S-HT-500). Amorphous

![Figure 3. XRD pattern of standard anatase [21] and rutile [22], TiO\(_2\)-A, TiO\(_2\)-S, TiO\(_2\)-A-HT, TiO\(_2\)-S-HT, TiO\(_2\)-A-HT-500, and TiO\(_2\)-S-HT-500 with anatase and rutile structures.](image3)

![Figure 4. Photoluminescence spectra of synthesized TiO\(_2\) particles before and after calcined process for (a) aggregate and (b) spherical bel particles.](image4)
phase in uncalcined samples provide higher defect density, i.e. surface defects, resulting higher photoluminescence (PL) intensity [23-25]. Figure 4 shows PL spectra of synthesized TiO$_2$ before and after calcination for both aggregate and spherical bcl particles. The dash-line is the limit of long wave pass filter and the straight-line represented the excitation laser line at 405 nm. The calcination process transforms amorphous titania phase into anatase crystalline phase decreasing defect density (see Figure 2 and 3). Thus, samples after calcination provide lower PL intensity due to the decrease of defects [23-25]. In TiO$_2$-S-HT-500 samples, we observed many sharp peaks with ordered spacing around 600 cm$^{-1}$ that corresponds to the phonon coupling of E$_{g}$(3) vibrational normal modes of anatase in electronic transition (see Figure 4b) [26].

**N$_2$ physisorption measurements were conducted to characterize surface properties of synthesized particles, i.e. the specific surface areas, pore volume, and pore diameter. The BET and BJH analysis results are presented in Table 1. In general, the specific surface area of the particles increases after alkaline hydrothermal treatment. For spherical particles, this is attributed to the increase of pore volume and the decrease of pore diameter. On the other hand, this is only attributed to the decrease of pore diameter for aggregated particles. This result is in a good agreement with the SEM analysis where alkaline hydrothermal treatment modifies the surface of particles through swelling and phase segregation resulting lamellar morphology with higher surface area (see Figure 1c and 1d). The calcination process decreases the specific surface area of the parti-**

![Figure 5](image)

**Figure 5.** N$_2$ physisorption isotherm of synthesized TiO$_2$ particles (a) TiO$_2$-A-HT-500, and (b) TiO$_2$-S-HT-500.

**Table 1.** N$_2$ Physisorption Analysis Results of Synthesized TiO$_2$ particles

| Sample          | Specific surface area ($m^2g^{-1}$)$^a$ | Pore volume ($cm^3g^{-1}$)$^b$ | Pore diameter (nm)$^b$ |
|-----------------|----------------------------------------|--------------------------------|------------------------|
| TiO$_2$-A       | 211                                    | 0.384                          | 7.25                   |
| TiO$_2$-A-HT    | 253                                    | 0.292                          | 4.62                   |
| TiO$_2$-A-HT-500| 119                                    | 0.214                          | 7.17                   |
| TiO$_2$-S       | 82                                     | 0.181                          | 17.45                  |
| TiO$_2$-S-HT    | 337                                    | 0.317                          | 7.53                   |
| TiO$_2$-S-HT-500| 74                                     | 0.134                          | 7.22                   |

$^a$) Multi-point BET

$^b$) BJH pore size distribution adsorption results
cles. This is mainly due to the decrease of pore volume for spherical particles and the increase of pore diameter for aggregated particles. Figure 5a and 5b show the N₂ physisorption isotherms of TiO₂-A-HT-500 and TiO₂-S-HT-500 samples, respectively. According to Figure 5a and 5b, we suggest the synthesized TiO₂ particles has a type IVa isotherm where capillary condensation is accompanied by hysteresis that starts to occur for pores larger than 4 nm in diameter [27]. In this isotherm, we can also observe the characteristic of type H3 hysteresis loop that occurs due to the presence of the non-rigid aggregates of plate-like particles (i.e. particles with lamellar morphology) but also if the pore network consists of macropores which are not completely filled with pore condensate (i.e. continuous v-groove or open channel networks) [27]. Thus, this type of isotherm supports the presence of bcl morphology on the particle surfaces [13].

Although the particles growth mechanism is still obscure, we proposed a general pathway based on bcl silica formation mechanism [13] and this result, to transform oxide particle with any shapes to bcl morphology (see Figure 6). Here, we demonstrated the transformation of aggregated and spherical TiO₂ particles to bcl morphology via alkaline hydrothermal route (see Figure 1c and 1d). This process shows a significant increase in specific surface area, especially for spherical particles. The specific surface area for calcined samples, both aggregated and spherical particles, is still higher with respect to state-of-the-art TiO₂ nanoparticles for photocatalyst, Degussa/Aeroxide P25 (S_{BET} = 35-65 m²g⁻¹) [28-30], despite of their microscopic size.

4. Conclusions

A simple and effective strategy has been demonstrated to transform aggregated and spherical particles to bcl morphology via alkaline hydrothermal route. We proposed this strategy as a general pathway to transform the particle surface with any shape to have bcl morphology. This strategy succeeded to achieve TiO₂ particles with a higher specific surface area than the state-of-the-art TiO₂ nanoparticles, Degussa/Aeroxide P25, despite of their microscopic size. This proposed synthesis strategy will open a new perspective in the development of mesoporous materials with open channel pore structure and their applications in a vast variety of fields.

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