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Facile Microwave Synthesis of 3D Flowerlike BiOBr Nanostructures and Their Excellent Cr^{VI} Removal Capacity

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A facile and fast microwave irradiation method has been developed to synthesize uniform well-defined 3D flowerlike BiOBr nanostructures through a self-assembly process. Cetyltrimethylammonium bromide (CTAB), which acts as both the bromide source and soft template, plays a critical role in the formation of 3D flowerlike BiOBr nanostructures.

Introduction

The controlled synthesis of nanostructures with unique size and shape-dependent properties has attracted increasing attention during the past decades. In particular, the design and fabrication of three-dimensional (3D) hierarchical nanostructures are of great interest and are being intensively pursued by virtue of their broad application in biomedical fields, energy storage, and catalysis.^[1-5] Recently, it was also found that 3D hierarchical nanostructures with porous and hollow structures exhibited excellent performance in the removal of heavy metal ions as highly effective adsorbents. Compared with the corresponding bulk materials and traditional adsorbents, hierarchical nanostructures possess improved removal capacity and a faster adsorption rate owing to their large surface area, special surface property, and so forth.^[6-11] Consequently, more research work focused on the fabrication and application of 3D hierarchical nanostructures. In the literature, most of the 3D nanostructures have been fabricated by a solvothermal strategy with the assistance of biomolecule, hard template, and microemulsion.^[1,12–15] Hence, it is still a challenge to develop simple and reliable synthetic methods for 3D nanostructures with the desired morphology.

As an important V–VI–VII ternary compound, bismuth oxybromide (BiOBr) has been extensively investigated be-

Different flowerlike BiOBr nanostructures could be obtained by varying the CTAB concentration. A possible formation process is discussed. The as-prepared flowerlike BiOBr nanostructures possessed a large surface area and exhibited an excellent removal capacity and fast adsorption rate for Cr^{VI} ions in a wide pH range.

cause of its unique electrical, magnetic, and optical properties, as well as its promising application in environmental remediation. Nanostructured BiOBr demonstrates not only excellent photocatalytic activity for NO oxidation, degradation of organic pollutants, and sterilization, but also an effective adsorption ability for organic dyes.^[16-19] Various Bi-OBr micro/nanostructures, which include nanoparticles,^[20] nanobelts, nanotubes,^[21,22] nanoplates, and microsheets,^[23,24] have been successfully fabricated by means of microemulsion, solvothermal, and sol-gel method. Apart from this, 3D hierarchically nanostructured BiOBr, which includes microspheres and microflowers, has also been synthesized by solvothermal method.^[15,25-28] Unfortunately. these fabrications led to characteristic crystalline structures. which thus showed significant differences in their performance. For example, almost all of the obtained 3D BiOBr hierarchical structures are closely packed and are dominated by large crystal size or irregular shape, which severely limit its potential adsorptive ability as well as its catalytic performance. In addition, the reported synthetic strategies suffered from the drawbacks of harsh conditions, long reaction times, and complexity. Thus, a facile and fast approach is highly desirable for the fabrication of diverse well-defined BiOBr hierarchical nanostructures. Recently, microwave irradiation method has been successfully developed to be a facile, fast, and mild synthetic route for the fabrication of various nanomaterials with controllable shape and size.^[29–32] It offers the advantage of uniform and dielectric heating without thermal gradient effects, which promote the reaction rate and led to an important advancement in the large-scale production of nanomaterials.

In this work, we report for the first time the successful synthesis of well-defined flowerlike BiOBr hierarchical nanostructures by a facile and fast microwave irradiation method. The obtained flowerlike BiOBr nanostructure possesses a highly specific surface area and large pore volume.

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As an example of its potential application in environmental remediation, the as-synthesized flowerlike BiOBr nanostructure was used as an adsorbent in water treatment and exhibited an excellent removal capacity for Cr^{VI} ions, which has not been reported in previous studies.

Results and Discussion

Structure and Morphology of BiOBr Nanostructures

The X-ray powder diffraction (XRD) pattern of the asprepared product (S1) is shown in Figure 1a. All of the diffraction peaks could be readily indexed to pure tetragonal phase BiOBr (JCPDS 73-2061) with a space group of P4 (No. 129) and lattice constants of a = 3.915 Å and c =8.076 Å. The diffraction peaks were remarkably broadened, which might be ascribed to the small crystalline size of the BiOBr crystals. The scanning electron microscopy (SEM) image (Figure 1b) of the BiOBr product revealed that the product is composed of a large quantity of uniform 3D flowerlike nanostructures with a diameter of 200 to 300 nm. The transmission electron microscopy (TEM) image is provided in Figure 1c for further investigation of the detailed morphology and structure of the flowerlike nanostructure, which showed that the entire structure is built from thin petals. These petals are loosely packed and connected to each other through the center to form flowerlike structures. A representative high-magnification TEM image taken from a piece of petal of the flowerlike structure is depicted in Figure 1d. It clearly revealed that these petals are constructed by very small nanoparticles (5-10 nm), which could explain the broadening of the XRD peaks. The corresponding selected-area electron diffraction (SAED) pattern illustrated that the as-prepared BiOBr nanostructure has a highly polycrystalline structure (Figure 1e). The energy dispersive X-ray (EDX) analysis also confirmed the formation of the BiOBr compound (Figure S1, Supporting Infor-



Figure 1. (a) XRD pattern, (b) SEM image, (c) TEM image, (d) high-magnification TEM image, and (e) SAED pattern of the flowerlike BiOBr nanostructures that were fabricated in the presence of 5 mM CTAB (S1).

mation). The ratio of the bismuth, bromide, and oxygen elements is approximately 1:1:1, which is in good agreement with the stoichiometry of BiOBr.

In this fabrication BiOBr was obtained under different CTAB concentrations (3-30 mM) as confirmed by its XRD patterns (Figure S2, Supporting Information). When the CTAB concentration was lower than 3 mm, no BiOBr product was collected. It was also found that CTAB played a unique role in controlling the morphology and size of the BiOBr nanostructure. Different flowerlike BiOBr nanostructures could be formed when the CTAB concentration was in the range of 3 to 16 mM (S2–S4). The typical SEM and TEM images of BiOBr products prepared in different CTAB concentrations are depicted in Figure 2. It is clear that the variation of CTAB concentration in this synthesis led to a dramatic change in the shape and size of the obtained BiOBr flowerlike nanostructures. When the CTAB concentration was 3 mm, SEM and TEM images revealed that well-defined flowerlike nanostructures with a uniform diameter of 350 nm were fabricated, which consisted of dozens of thin petals (S2, Figures 2a and 2b). With an increase in the CTAB concentration to 8 mm, the size of the flowerlike nanostructures decreased to about 150 nm in diameter (S3, Figures 2c and 2d). However, irregular flowerlike structures with fewer petals and a diameter of ca. 110 nm were obtained in the presence of 16 mM CTAB solution (S4, Figures 2e and 2f). When the CTAB concentration was higher than 16 mm, there was an obvious tendency to form bulk agglomeration (S5, Figure S3, Supporting Infor-



Figure 2. SEM and TEM images of BiOBr nanostructures synthesized in different CTAB concentrations: (a and b) 3 mM, S2; (c and d) 8 mM, S3; (e and f) 16 mM, S4.

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mation). This indicated that CTAB acted as not only the bromine source but also as a soft template and that proper CTAB concentration facilitated the assembly of petals into 3D flowerlike nanostructures with controllable shape and size through the interaction between the CTAB molecules and the petals, which is similar to the cases reported in the literature.^[9,33] In order to verify the role of CTAB, control experiments were carried out using KBr instead of CTAB. However, only platelike BiOBr nanostructures were obtained in the presence of different KBr concentrations under the identical reaction conditions (S6–S8, Figure S4, Supporting Information).

Possible Formation Process of Flowerlike BiOBr Nanostructures

BiOBr Nanostructures and CrVI Removal Capacity

In order to investigate the formation process of the flowerlike nanostructure further, time-dependent experiments were conducted. The SEM images of the products prepared in the presence of 5 mM CTAB at different reaction times are displayed in Figure 3. At the early stage (5 min, S9), the product was composed of BiOBr nanoplates together with partial flowerlike nanostructures (Figure 3a). After a 10 min microwave reaction, the amount of flowerlike nanostructures increased (S10, Figure 3b). As the reaction proceeded to 20 min, there was a tendency to form flowerlike nanostructures for the whole sample (S11, Figure 3c). Eventually, well-defined flowerlike nanostructures were obtained after a 30 min reaction (S1, Figure 3d). This process involved two stages. Nanopetals formed quickly in the initial stage, which was followed by a relatively slow aggregation of the petals into flowerlike nanostructures. In addition, the reaction temperature is an important parameter during the fabrication process of BiOBr flowerlike nanostructures. In this synthesis, different BiOBr flowerlike nanostructures (S1-S4) were fabricated at the same reaction temperature (110 °C). At low temperatures (30 or 60 °C), for instance, only large crystals with irregular morphology were obtained (S12 and S13, Figure S5, Supporting Information), which indicates that the formation of flowerlike nanostructures is affected by the reaction temperature. However, the influence of the reaction temperature is a complicated issue in the microwave synthesis and needs further detailed investigation.

In the literature, it was reported that CTAB might form spherical micelles, wormlike micelles, or even high-order phases. Generally, spherical micelles are dominant in a CTAB solution concentrated over its critical micelle concentration (CMC) and wormlike micelles predominate at higher concentrations.^[34,35] In the present synthesis, a dramatic variation in the size and shape of flowerlike BiOBr nanostructures might be due to the different micelle structures depending on the CTAB concentration. From the experimental results, a micelle-dependent model was proposed for the formation of flowerlike BiOBr nanostructures. As illustrated in Scheme 1, uniform spherical micelle structures were formed at low CTAB concentrations near CMC, which were surrounded by BiO+ ions generated from the bismuth precursor.^[2] Under microwave irradiation, BiO⁺ ions reacted with CTAB to form BiOBr nuclei. In the growth process that followed, 2D petals were preferentially formed depending on the intrinsic BiOBr layered structure.^[26] The petals then assembled to construct the welldefined flowerlike BiOBr nanostructure through the interaction between CTAB molecules and the petals. With the increase of CTAB concentration, wormlike micelles gradually predominated but these micelles break and transfer into spherical ones when the temperature is increased.^[36,37] Thus, it was imagined that more but smaller spherical micelle structures might be generated from the broken wormlike micelles at relatively high concentration under microwave heating, which leads to the formation of BiOBr flowerlike nanostructures with smaller size and fewer petals. However, the particles would be totally capped by CTAB molecules at very high CTAB concentration in the formation process of BiOBr, which would finally result in the agglomeration (S5). In fact, our present understanding of the formation of BiOBr flowerlike nanostructures is quite limited and further investigation is in progress.



Figure 3. SEM images of BiOBr products obtained at different reaction times: (a) 5 min, **S9**; (b) 10 min, **S10**; (c) 20 min, **S11**; and (d) 30 min, **S1**.



Scheme 1. Illustration of possible formation mechanism of flowerlike BiOBr nanostructures in the presence of different CTAB concentrations.

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Nitrogen Adsorption–Desorption Isotherm of BiOBr Nanostructures

A nitrogen adsorption-desorption isotherm measurement was conducted to further investigate the structural characteristics of the flowerlike BiOBr nanostructure obtained in a 5 mM CTAB solution (S1). As shown in Figure 4a, it displayed a type IV isotherm with a type H3 hysteresis loop at high relative pressures between 0.8 and 1.0, which indicates the presence of mesopores (2–50 nm).^[38] Moreover, the hysteresis loop shifted approach $p/p_0 \approx 1$, which demonstrates the presence of large macropores (> 50 nm).^[39] This was further confirmed by the corresponding Barrett-Joyner-Halenda (BJH) pore diameter distribution in which the peaks for both mesopores and macropores up to 100 nm were found (Figure 4b). Considering the observed morphology and structure of the product, the smaller pores with sharp peaks at about 2 and 6 nm might be generated during the crystal growth process, whereas the larger pores with a wide pore diameter distribution could be attributed to the space between the intercrossed BiOBr petals. The Brunauer-Emmett-Teller (BET) specific surface area of S1 was estimated to be $63.5 \text{ m}^2\text{g}^{-1}$, which is much larger than that of platelike BiOBr nanostructures (33.6 m² g⁻¹, Figure S6, Supporting Information). The large BET surface area, ideal pore size distribution, and pore volume (0.19 m³ g⁻¹) make the as-obtained BiOBr flowerlike nanostructure a promising candidate as an adsorbent for heavy metal ion removal in water treatment.



Figure 4. (a) Nitrogen adsorption–desorption isotherm and (b) pore size distribution of the flowerlike BiOBr nanostructures obtained in 5 mM CTAB solution (S1).

Adsorption Capacity for Cr^{VI}

The time-dependent optical absorption spectra of a $K_2Cr_2O_7$ solution with an initial Cr^{VI} concentration of 21 mg L⁻¹ in the presence of 20 mg of flowerlike BiOBr nanostructure S1 is shown in Figure 5a. With the increase in the adsorption time, the absorption peak of the $K_2Cr_2O_7$ solution intensely decreases. The removal efficiency was about 94% after 2 h of adsorption, which was much higher than that of platelike BiOBr nanostructures (Figure S7, Supporting Information). The time-dependent Cr^{VI} concentration variation curve over the flowerlike and platelike BiOBr products is given in Figure 5b. It indicated that the flowerlike BiOBr nanostructure exhibited a very fast adsorption rate for Cr^{VI} ions at the initial stage and that 79%

of the Cr^{VI} ions could be adsorbed within 15 min. The $K_2Cr_2O_7$ removal capacity of flowerlike and platelike Bi-OBr was measured to be 56.2 and 31.4 mg g⁻¹, respectively, which is much higher than that of the reported nanoscaled adsorbents.^[9,40–42] It was evident that the flowerlike BiOBr nanostructure showed superior removal capacity, which should be mainly ascribed to its highly specific surface area and loosely packed structure.



Figure 5. (a) Time-dependent optical absorption spectra of $K_2Cr_2O_7$ solution with an initial Cr^{VI} concentration of 21 mg L^{-1} in the presence of 20 mg of flowerlike BiOBr nanostructure **S1**; (b) time-dependent Cr^{VI} concentration variation over different BiOBr nanostructures.

More importantly, the superior removal capacity of the as-obtained flowerlike BiOBr nanostructure could be realized in a wide pH range. The CrVI removal capacity of the flowerlike BiOBr nanostructure S1 at various pH values is illustrated in Figure 6. It was found that Cr^{VI} ions could be effectively removed over a wide pH range from 3 to 11 and that the removal capacity decreased when the pH value was below 3 or above 11. It was proposed that the removal capacity at different pH values was strongly related to the surface charge of BiOBr as well as the Cr^{VI} species present in solution. The zeta potential of flowerlike BiOBr nanostructures was estimated to be 30.6 mV in deionized water (Figure S8, Supporting Information), which illustrated the existence of positive charge on the BiOBr surface. In the $K_2Cr_2O_7$ solution, the HCrO₄⁻ and CrO₄²⁻ ions with negative charge are the dominant species of CrVI in the pH range of 3 to 11.^[43,44] It is believed that the highly efficient removal of Cr^{VI} in the pH value of 3 to 11 is probably due to the strong coulombic attraction between the positively charged BiOBr nanostructures and the negatively charged Cr^{VI} ions (HCrO₄⁻ or CrO₄²⁻). The poor adsorption capacity at low pH values (< 3) is mainly owing to the formation of the uncharged H₂CrO₄ species, which cannot be adsorbed efficiently. Large quantities of OH⁻ ions in the high pH value (>11) solutions would compete with the Cr^{VI} species for adsorption sites on the surface of BiOBr, which would result in the decrease in efficiency of Cr^{VI} removal. Generally, most reported absorbents reached their maximum adsorptive capacity of heavy metals only at the optimal pH value.^[40,41,45,46] Hence, we believe that the flowerlike BiOBr nanostructure with its remarkable removal capacity in a wide pH range would be a significant potential absorbent, which could be used in practical water treatment.

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Figure 6. Effect of pH on the Cr^{VI} ions removal in the presence of 20 mg of flowerlike BiOBr nanostructure S1.

Conclusions

In summary, a facile and fast microwave irradiation method has been developed for the first time to synthesize well-defined 3D flowerlike BiOBr nanostructures by using CTAB as the bromide source and soft template. In this fabrication, CTAB played a crucial role in directing the construction of 3D hierarchical BiOBr nanostructures. A possible formation process of flowerlike BiOBr nanostructures in different CTAB concentrations was discussed. The assynthesized BiOBr 3D flowerlike nanostructure possessed a large BET surface area and exhibited excellent removal capacity and fast adsorption rate for CrVI in a wide pH range, which demonstrated that it could be used as an ideal material for the removal of CrVI ions from water. It is also expected to be useful in many other applications, such as sterilization, degradation of organic pollutants, and so forth.

Experimental Section

Chemicals: Bismuth nitrate pentahydrate [Bi(NO₃)₃·5H₂O] was purchased from Tianjin Fuchen Chemical Reagents Factory (China). Mannitol was purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). K₂Cr₂O₇, KBr, and cetyltrimethylammonium bromide (CTAB) were purchased from Aladdin (Shanghai, China). All of the reagents were analytical grade and were used directly without further purification.

Preparation of BiOBr Nanostructures: In a typical synthesis, $Bi(NO_3)_3$ - $5H_2O$ (0.485 g, 1 mmol) was dissolved in mannitol (30 mL, 0.1 M) that contained CTAB (5 mM). The mixture was stirred and sonicated until all of the chemicals were well dispersed. The mixture for 27 min in a microwave reactor (XH-300A, Beijing Xianghu Technology Co., Ltd.), irradiation power was 500 W. After microwave treatment, the mixture was cooled to room temperature. A white precipitate was obtained by centrifugation and washed with deionized water and ethanol five times. Finally, the product was dried in a desiccator for a few days for further characterization. Other samples of BiOBr nanostructures were also prepared under identical conditions by changing the Br source, reaction time, and reaction temperature. The detailed experimental parameters are listed in Table 1.

Table 1. Experimental conditions for the preparation of BiOBr nanostructures.

	Br precursor	<i>С</i> [mм] ^[а]	Temp. [°C]	Time [min]
S1	СТАВ	5	110	30
S2	CTAB	3	110	30
S3	CTAB	8	110	30
S4	CTAB	16	110	30
S5	CTAB	30	110	30
S6	KBr	3	110	30
S7	KBr	5	110	30
S8	KBr	30	110	30
S9	CTAB	5	110	5
S10	CTAB	5	110	10
S11	CTAB	5	110	20
S12	CTAB	5	30	30
S13	CTAB	5	60	30

[a] The concentration of Br source.

Characterizations: X-ray powder diffraction (XRD) was carried out with a Bruker axs D8 Discover (Cu- $K_a = 1.5406$ Å) instrument at a scan rate of 2° min⁻¹ in the 2θ range from 10 to 70°. Scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) images were taken with a Hitachi S4800 scanning electron microscope operating at 5.0 kV. TEM and selected-area electron diffraction (SAED) images were recorded with a Philips Tecnai 20 electron microscope by using an accelerating voltage of 200 kV. Samples for TEM analysis were prepared by drying a drop of nanocrystal dispersion in absolute ethanol on carbon-coated copper grids. The Brunauer-Emmett-Teller (BET) specific surface area of the powders was analyzed by nitrogen adsorption with a Micromeritics ASAP 2020 nitrogen adsorption apparatus (USA). All of the asprepared samples were degassed at 100 °C for 4 hours prior to the nitrogen adsorption measurements. The zeta potential for the product was determined with a Malven ZEN3690 zeta potential analyzer. The pH of the K₂Cr₂O₇ solution was adjusted with 1 M HCl or 2 м NaOH and measured with a pH meter (Sartorius).

Adsorption Experiment of BiOBr Nanostructures: The initial Cr^{VI} solution (21 mg L⁻¹) was prepared by dissolving $K_2Cr_2O_7$ (60 mg) in deionized water (1 L) at room temperature. BiOBr powders (20 mg) were added to the prepared Cr^{VI} solution (20 mL) whilst stirring. At each given time interval, the suspension was sampled (2 mL) and centrifuged to remove the BiOBr powders. The concentration of $K_2Cr_2O_7$ during the adsorption was measured with a Shimadzu UV2800 spectrophotometer. All of the measurements were carried out at room temperature.

Supporting Information (see footnote on the first page of this article): EDX spectrum of S1, XRD patterns of S2 to S8, SEM images of S5 to S8, S12, and S13, nitrogen adsorption–desorption isotherm of S7, time-dependent optical adsorption spectra of $K_2Cr_2O_7$ solution in the presence of S7, and zeta potential of S1.

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Nanostructures by Microwave

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Facile Microwave Synthesis of 3D Flowerlike BiOBr Nanostructures and Their Excellent Cr^{VI} Removal Capacity

Keywords: Bismuth oxybromide / Nanostructures / Templated synthesis / Microwave chemistry / Chromium

Well-defined 3D flowerlike BiOBr nanostructures have been successfully synthesized by a facile and fast microwave irradiation method by using cetyltrimethylammonium bromide as both the bromide source and soft template. The as-prepared flowerlike BiOBr nanostructures possess a large surface area and exhibit an excellent removal capacity and fast adsorption rate for Cr^{VI} ions in a wide pH range.

