Solvothermal Synthesis and Characterization of Nest-shaped BiOCl

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Abstract. BiOCl with nest morphology was prepared successfully. By the condition that the ratio of BiCl\textsubscript{3} to urea is 1:2 and with a simple solvothermal route. The morphology and composition of BiOCl were studied by X-ray diffractometer (XRD), scanning electron microscope (SEM), and UV-vis spectrophotometer methods. SEM result indicates that the sample exhibited nest-shaped structures. XRD results showed that the synthesized sample were pbfc1 layered structure BiOCl with good crystallinity. UV−vis spectra reveal that the band gap energy of the BiOCl was 3.57 eV.

1. Introduction
Since the last decade, to solve the problem on photocatalytic degradation of organic pollutants and solar energy conversion, scientists have looked for many ways. Among them, photocatalysts has remarkable effect. Due to BiOCl has a special layered structure, good optical properties and high photocatalytic activity and stability, it has attracted great attention from scientists. The quintuple layers of BiOCl from the tetragonal structure of P4/nmm space group, with [Cl–Bi–O–Bi–Cl] stacked together, and the nonbonding interaction through the Cl atoms along the c-axis with lattice constants a=b=3.89 Å and b=7.37Å. Due to the V-VI-VII semiconductors of BiOCl, the unique wide band gap of BiOCl is around 3.6 eV, and it often used on photocatalytic degradation with ultraviolet (UV) light detection method, split solar water, fix nitrogen, photocatalytic treatment of sewage and pure gas with pollution [1-10]. In a word, BiOCl is a kind of photocatalytic material with good development prospect.

In this work, under the premise that the total volume of EG is unchanged, we have successfully prepared nest-shaped BiOCl nanometer material through a simple solvothermal method.

2. Experimental
2.1. Materials and Reagents
Bismuth chloride (BiCl\textsubscript{3}, A.R.) was gained from Sinopharm Group Chemical Reagent Co., Ltd. Ethylene glycol (EG) and Urea (H\textsubscript{2}NCONH\textsubscript{2}, A.R.) was obtained from Shanghai Titan Scientific Co., Ltd. All the reagents used in the experiment were in analytical grade and do not do more processing in the experiment.

2.2. Preparation of Powder BiOCl
In the experiment, prepare a 150ml beaker, according to take 0.630g of BiCl3 and 40 mL ethylene glycol (EG) in the beaker, stirring them to make it mixed at 28°C. In half an hour, according to take 0.240g urea in the beaker. When spread evenly, transfer the solution to a 50ml of Teflon-lined stainless steel
autoclave. Rotate the autoclave to make it sealed, then put it in a 160°C oven for 8 h. When the time is up, remove the autoclave from the oven and let it cool naturally. Then, the sample was transferred to a 50 ml centrifuge tube, poured out the supernatant after centrifugation, wash it with ultra-pure water for 3 times and ethanol for 3 times. Finally, put it in a 60°C oven make it dried.

2.3. Characterization
With X-ray diffractometer (XRD, Shimadu, Japan), we can obtain the Powder BiOCl is made of which phase composition and the crystalline structure, set up the voltage of the XRD at 40 kV and the current at 200 mA. At the same time, use Cu-Kα as radiation. By scanning electron microscopy (SEM, JEOLJSM-7001F, Japan), the morphologies and particle sizes of the sample can be observed. UV–vis spectrophotometer (UV-3600, Shimadzu, Japan) can detect UV–vis diffused reflectance spectra (DRS), using BaSO₄ as a reflectance standard.

3. Results and Discussion

3.1. XRD Analysis
Results of powder BiOCl shown in Figure 1 below. The diffraction peaks of the tetragonal phase of BiOCl (JCPDS NO.06-0249) corresponds to the powder BiOCl in this experiment. Every peak is very pure of the result and we can come to a conclusion that the purity of powder BiOCl is superb. In addition, all diffraction patterns have narrow and strong diffraction peaks and no hybrid peaks, indicating that the crystallinity of the product is very good.

![Figure 1. XRD result of BiOCl architectures.](image)

3.2. Structure and Morphology
Using SEM detect the morphology of the sample. The SEM image of Figure 2a is a overall result typical low-magnification, which displays that the dispersion is good and the average particle size is 3~4 um. Figure 2b shows that the spheres are nest-shaped and exhibit a hierarchical structure. Figure 2c displays that the layered structure is composed of a number of self-assembled nanocrystals. In addition, probably due to Ostwald ripening, the BiOCl has a very smoothly surface of the sheets assembled.
3.3. Optical Absorption Property
Using the UV-vis give the diffuse reflectance spectra of the powder BiOCl sample. Figure 3a presents that in the visible light region of 300-370nm, the sample has great absorption, indicates that the BiOCl exhibit excellent abilities of harvesting Ultraviolet light. Figure 3b show the Tauc plots-(\(\alpha h\nu\))^2 versus the energy of absorbed light (\(h\nu\)), where \(\alpha\), \(h\) and \(v\) represent the absorption coefficient of the sample, Planck's constant and frequency respectively [11]. The Eg value of as-obtained BiOCl sample can be evaluated to be about 3.57 eV.

4. Conclusion
In conclusion, a facile and template/surfactantfree method to synthesize BiOCl with a simple solvothermal route have developed. In the future, it can be used in photocatalytic areas and will has a good results. Furthermore, by properly changing the synthetic conditions, BiOCl with different morphologies and related inorganic materials could be obtained.

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