Synthesis of A Marigold-like BiOCl Nanocomposite With Enhanced Photocatalytic Activity

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Abstract: The marigold like Bismuthoxychloride/polystyrene (BiOCl/PS) nanocomposite was successfully synthesized by a facile method. XRD results revealed that the prepared materials were crystalline in nature. Morphological study evidenced that the prepared BiOCl catalyst consist of flakes with a diameter of around 1-2 μm. The bandgap energy of BiOCl/PS nanocomposite was found to be 3.2 eV which is well supported to pure BiOCl. The photocatalytic degradation experiment was performed by degradation of methylene blue dye. The obtained results show that the degradation rate by flower like BiOCl/PS nanocomposite was 85% and for pure BiOCl was 25% respectively which confirms flower like BiOCl/PS nanocomposite is highly efficient photocatalyst.

INTRODUCTION

A composite of inorganic particles with polymers is an interesting research subject because of the important potential applications in wastewater remediation. The polymer nanocomposite has been successfully used for various stable inorganic colloids free from aggregation, such as titanium oxide, zinc oxide, magnetic and metal nanoparticles [1, 2]. The Sillén family compounds with unique layered structure have often been investigated as photo catalyst [3]. BiOCl with large band gap (Eg = 3.6 eV) does not exhibit any photocatalytic activity under visible light, but it was reported that it reveals excellent photocatalytic activity for degradation of organic dyes under UV irradiation [4, 5]. As a matter of BiOCl/PS nanocomposite, the former is used as a photo catalyst because it has drawn its considerable attention for its unique and excellent optical, electrical, magnetic properties and photocatalytic performance [6, 7]. In the present work, BiOCl/PS nanocomposite was prepared by a facile method. A systematic study on effect of visible light irradiation on optical and structural properties of prepared nanocomposite has been carried out. As prepared material were characterized by XRD, Scanning electron microscope (SEM) and Transmission electron microscope (TEM) and UV-Vis spectroscopy.

EXPERIMENTAL

Preparation of marigold like Bismuthoxychloride: The flower like bismuthoxychloride was prepared using polyvinylpyrrolidone (PVP) and Bismuth trichloride. 1.50 gm of polyvinylpyrrolidone (PVP) is added to 60 ml of distilled water in a 100 ml beaker. The solution was stirred and then 1.42 gm of bismuth trichloride was added to the solution with vigorous stirring upto 10-15 minutes. pH was adjusted upto 9.0 for precipitation by adding ammonia solution. The solution then allowed settling for half an hour until precipitation completes. After that the solution was filtered and the precipitates are washed with distilled water then precipitates were dried at 80 °C for 24 hours [8]

Preparation of BiOCl/PS nanocomposite: Polystyrene (PS) was dissolved in dichoromethane in 25 ml glass beaker. The solution was magnetically stirred continuously in order to uniform dissolution of polymer then appropriate amount of as prepared BiOCl particles was added to the solution. The prepared viscous solution
containing BiOCl was poured over a flat bottomed petridish. The solvent was allowed to evaporate by keeping it for 12 – 24 hours in a dry atmosphere and then film was peeled off from petridish for further characterization.

**XRD ANALYSIS**

Figure 1 shows a typical XRD pattern of as-prepared BiOCl and BiOCl/PS nanocomposite, with sharp peaks indicates the good crystal quality. All the diffraction peaks can be indexed to the tetragonal structure of BiOCl (JCPDS Card No. 85-0877). No diffraction peaks of Bi are observed, indicating complete conversion of Bi into BiOCl. Compared with the reference diffraction pattern (JCPDS Card No. 85-0877), the diffraction peak intensity of the (110) plane is relatively stronger than that of other planes. This probably relates to the growth orientation of the nanopetals of the flower structure. In XRD pattern of BiOCl/PS nanocomposite, the intensity of (110) peak was reduced.

**FIGURE 1.** X-ray diffraction pattern of as prepared pure BiOCl and BiOCl/PS nanocomposite

**SEM AND TEM ANALYSIS**

The surface morphology of marigold like BiOCl and its nanocomposite was studied under SEM and TEM represented in Fig. 2 and Fig. 3. Figure 2 (a) is a SEM image of BiOCl, revealing that the sample consist of two-dimensional (2D) flakes. The flakes can further assemble into a marigold flower like nanostructure shown in Fig. 2(a). The diameter of the whole nanoflower is around 1-2 μm. TEM results Fig. 2(b) further confirms that BiOCl consists of sheets stacked together to form flower like structure.

**FIGURE 2.** (a) SEM image, (b) TEM image of as prepared BiOCl
The SEM image of marigold BiOCl/PS nanocomposite is represented in Fig. 3. The nanocomposite exhibits uniform distribution morphology and shows that the BiOCl have good interfacial adhesion with matrix. The BiOCl nanosheets stack together to constructs aggregates. A more detailed insight of the BiOCl/PS nanocomposite was examined by TEM measurements (Fig 3(b)). Some nanoparticles are still attached with the sheets, indicating the strong interactions between the BiOCl and the sheets [9,10].

**UV-VISIBLE ANALYSIS**

UV-visible study shows the optical absorption property of BiOCl thin film. In Fig. 4, it is showed that the BiOCl nanocomposite exhibits strong light absorption at wavelength lower than 380 nm. The bandgap energy (Eg) of BiOCl nanocomposite, as estimated from the corresponding plot of \((\alpha h\nu)^{1/2}\) versus photon energy \((h\nu)\) (inset of Fig. 4) is about 3.2eV which is beneficial for photocatalytic degradation of pollutants in visible range.

**PHOTOCATALYTIC ACTIVITY**

To evaluate the photocatalytic activity of the prepared marigold like BiOCl/PS nanocomposite, UV-visible characteristics of methylene blue dye as a pollutant were investigated under visible light irradiations. In this experiment different sets of 100 to 500 mg of the catalyst BiOCl were added in 100 ml of 1 x 10^{-5} M methylene blue (MB) solution. Before irradiation, the suspensions were magnetically stirred for 10-15 minutes. Then the solution was exposed to visible light with constant stirring. After given time interval of 10 min, 5ml of the aliquot was pipette out and centrifuged for 2-3 min. The absorbance of the filtrates were analysed at maximum absorbance of 653nm during the photodegradation process. Under the visible light irradiation, the strong absorption peak of MB
solution at 653nm steadily decreased with increasing light irradiation time and the blue colour of the solution changes gradually to colourless showing that the MB solution has been degraded completely. The degradation efficiency of pure BiOCl and marigold like BiOCl/PS (see Fig. 5) was compared for different amount of catalysts dosage within the time interval of 30 min.

![Irradiation time 30 min](image)

**FIGURE 5.** Photocatalytic activity of flower like BiOCl and Pure BiOCl.

Photocatalytic performance of the product was evaluated by photodegradation process of MB from the aqueous solutions. The degradation efficiency is calculated by eq:

% Degradation Efficiency = \( \frac{(C_0 - C)}{C_0} \times 100 \)

Where, \( C_0 \) is the initial concentration of dye and \( C \) is the concentration of dye after treatment at different times, \( t \) (0-30min). The experimental results showed that the maximum degradation efficiency of marigold like BiOCl/PS within 30 min is 85% whereas for pure BiOCl the degradation efficiency was about 25%. This confirms that the as prepared BiOCl was highly efficient in degradation of MB within 30 min than of pure BiOCl, (see Fig. 5).

**CONCLUSION**

The marigold like BiOCl/PS catalyst was successfully synthesized which shows high photocatalytic activity than the pure BiOCl. In XRD, the sharp peaks reveal a high degree of crystallization for the products. SEM images indicates the prepared BiOCl catalyst comprise of flakes with a diameter of about 1-2 μm. The bandgap energy \( (E_g) \) of BiOCl/PS nanocomposite was calculated to be 3.2eV. The comparison experiments of photoactivity between pure BiOCl and marigold like BiOCl/PS shows that the degradation rate in case of marigold like BiOCl/PS was 85% and 25% for pure BiOCl which confirms the synthesis of highly efficient BiOCl.

**REFERENCES**