

The Role of Aqueous Inorganic Ions in Modulating the Photocatalytic Activity of Faceted BiOCl

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Abstract:

The coexistence of inorganic ions in aquatic environments plays a crucial role in influencing photocatalytic performance. In this study, well-defined BiOCl nanosheets, characterized by dominant {001} and {010} facets, were employed as a model photocatalyst to investigate the effects of various inorganic ions on the photocatalytic degradation activity of faceted photocatalysts. The results indicated that the introduction of inorganic ions significantly modulated the photocatalytic degradation rate of sulfamethoxazole (SMX) over the faceted BiOCl photocatalyst. BiOCl exhibited facet-dependent photocatalytic activity for SMX degradation, with the {001} facet demonstrating superior performance compared to the {010} facet under both cationic and anionic conditions. This study highlights the importance of facet-dependent synergy in the rational design of photocatalysts for the effective degradation of persistent pollutants in practical applications.

Keywords: BiOCl Photocatalysis, Facet-Dependent Reactivity, Inorganic Ion Modulation

1. Introduction

The global challenges of energy sustainability and environmental pollution have intensified the search for effective solutions in recent years.^[1] Among the promising approaches to address these issues, semiconductor-based photocatalysis has emerged as a vital technology for both energy conversion and the degradation of organic pollutants.^[2,3] Semiconductor materials can absorb light to drive chemical reactions. Since the photocatalytic reaction primarily occurs at the photocatalyst surface, the reactivity and efficiency of photocatalysts are greatly influenced by their surface atomic structure, which determines

critical processes such as charge carrier transfer, the availability of reactive sites, and the adsorption/desorption of reactants.^[4,5]

Different crystal facets of semiconductor materials exhibit distinct electronic and chemical properties, which can significantly affect their photocatalytic activities. For instance, titanium dioxide (TiO₂), bismuth vanadate (BiVO₄), and bismuth oxychloride (BiOCl) have demonstrated crystal facet-dependent photochemical reactions.^[6-8] Particularly, BiOCl is of great interest due to its unique layered structure, which consists of alternating [Bi₂O₂] units interspersed with halide layers.^[9] This architecture facili-

tates the formation of built-in electric fields that promote efficient charge separation and allows for tuning of surface characteristics to achieve optimized photocatalytic performance. For instance, Zhang et al. have shown that BiOCl with predominantly exposed {001} facets exhibits superior photocatalytic activity for the degradation of organic pollutants under UV irradiation compared to those with {010} facets. Increasing evidence supported the crucial role of crystal facets in a modulating the photocatalytic performance for pollutants degradation.^[10]

However, in practical applications, wastewater often contains a complex mixture of organic contaminants alongside a variety of inorganic ions, such as Na^+ , Ca^{2+} , Mg^{2+} , Cl^- , SO_4^{2-} , and NO_3^- . These ions can significantly influence the photocatalytic degradation processes, as they may alter the adsorption processes on the catalyst surface, affect the generation of reactive oxygen species (ROS), and even modify the surface electronic properties of the photocatalyst.^[11,12] Despite the importance of this interaction, there has been limited investigation into how different inorganic ions affect the performance of BiOCl photocatalysts, particularly those with distinct crystal facets.

Herein, we synthesized BiOCl nanocrystals with exposed {001} and {010} facets to investigate the effects of different inorganic ions on the photocatalytic degradation activity of sulfamethoxazole (SMX) over BiOCl photocatalyst. Our findings will deepen the understanding of the structure-activity relationship in photocatalysis and provide valuable insights for designing advanced photocatalysts for environmental remediation.

2. Experimental Section

2.1 Materials and reagents

All chemicals used in this study were obtained from Sino-pharm Chemical Reagent Co., Ltd. (Shanghai, China) and were of analytical grade, used without further purification. All solutions were prepared using ultrapure Milli-Q water with a resistivity of $18.2 \text{ M}\Omega \cdot \text{cm}^{-1}$ (Millipore).

2.2 Preparation of faceted BiOCl photocatalysts

Faceted BiOCl nanocrystals were synthesized following a previously established method with slight modifications.^[13] In a typical synthesis, 1.46 g of $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ and 0.224 g of KCl were dissolved in 50 mL of distilled water at room temperature under continuous stirring. The pH of the solution was adjusted to 6.0 using 1 M NaOH. After 30 minutes of stirring, the mixture was transferred into an autoclave, which was then heated to 160 °C for 24 hours

before cooling down to room temperature. The resulting precipitate was collected and thoroughly washed with de-ionized water and ethanol multiple times, then air-dried. The BiOCl nanosheets synthesized at pH 6, featuring predominantly the {010} crystallographic facet, were designated as BOC-010. The BiOCl nanosheets exposing the {001} facet were synthesized under identical conditions but without pH adjustment, designated as BOC-001.

2.3 Photocatalytic degradation experiments

To evaluate the photocatalytic efficiency of the prepared catalysts, degradation experiments of sulfamethoxazole (SMX) were conducted using a 250 W Xe lamp to provide UV light by equipping it with $\lambda > 400 \text{ nm}$ cutoff filter. In the photocatalytic degradation experiments, the concentration of SMX was set at 5 mg/L, the concentration of the photocatalyst was 1.0 g/L, and the total reaction volume was 50 mL. The mixture was stirred continuously in the dark for 30 minutes to achieve adsorption-desorption equilibrium prior to the light irradiation. To maintain a stable temperature of 25 °C during the reaction, a circulating water system was employed. At certain time intervals, 2 mL aliquots were withdrawn from the reaction mixture. After centrifugation to separate the catalyst, the pollutant concentration was determined using high-performance liquid chromatography (HPLC, Prominence, Shimadzu) equipped with a C18 column and a UV detector set to a wavelength of 265 nm. The mobile phase consisted of a mixture of 0.1% acetic acid- H_2O and methanol in a volume ratio of 47.5:52.5, with a flow rate maintained at 0.3 mL/min. Additionally, to investigate the influence of various inorganic anions on the photocatalytic performance of the faceted BiOCl catalysts, 100 mM solutions of specific inorganic anions, including Cl^- , HCO_3^- , SO_4^{2-} , NO_3^- , K^+ , Ca^{2+} , and Mg^{2+} , were introduced into the solution in separate experimental setups.

3. Results and Discussion

3.1 Morphological characterization of faceted BiOCl nanocrystal

BOC-001 and BOC-010 were synthesized through a simple hydrothermal method, with the crystal facets controlled by adjusting the pH of the solution. Scanning electron microscopy (SEM) was used to examine the morphological characteristics of the synthesized BiOCl nanocrystals. As shown in Figure 1a, BOC-001 sample displayed large sheet-like structures with widths ranging 2~3 μm and thicknesses 90~250 nm. Based on the tetragonal symmetry of BiOCl nanocrystal, the top and bottom

surfaces of the BOC-001 sample are identified as {001} facets, while the lateral surfaces correspond to the {110} and {100} facets.^[14] Geometric calculations estimated the proportion of {001} facets to be approximately 76% for BOC-001. Figure 1b illustrated that the synthesized BOC-010 sample also displayed a well-defined nanosheet structure, with estimated widths and thicknesses of 200~700 nm and 20~50 nm, respectively. The surfaces of BOC-010 are primarily enclosed by {010} facets, with an estimated surface exposure of about 81% for these facets.

The {001} facet features a closed structure, with the outermost layer composed of Cl, O, or Bi atoms, whereas the {010} facets demonstrate an open-channel arrangement, alternately displaying Cl, Bi, and O atoms.^[15] The arrangement of surface atoms and their coordination in semiconductors are crucial in determining their electronic and physicochemical properties. The ability to manipulate the exposed facets of BiOCl nanocrystal allows for a detailed study of their photocatalytic performance in relation to facet orientation.

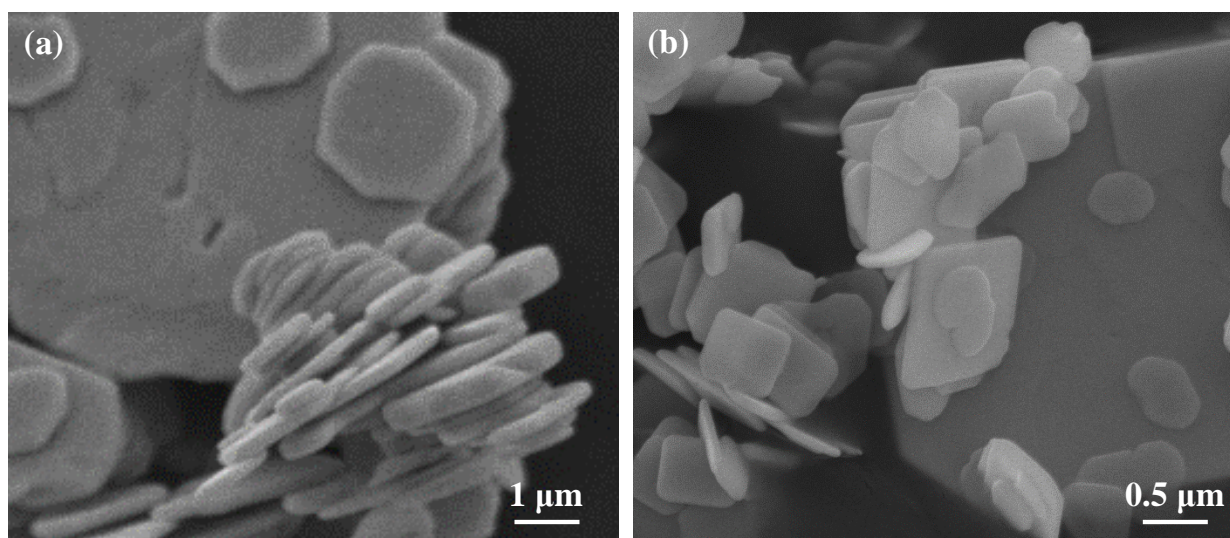


Figure 1. SEM images of (a) BOC-001 and (b) BOC-010 nanocrystals.

3.2 Effect of cations on the photocatalytic degradation of SMX

The photocatalytic degradation of SMX over faceted BiOCl nanocrystals was systematically evaluated. As shown in Figure 2a, BiOCl nanocrystals could promote SMX degradation. After 30 minutes of exposure to UV light, the degradation efficiencies of SMX reached approximately 98.1% over BOC-001 and 91.0% over BOC-010. These results indicate that BOC-001 is markedly more effective in photocatalytic degradation of SMX, likely due to the higher surface energy of {001} facets, which could facilitate more efficient charge separation and reactivity.

Further investigations into the effects of various cations, e.g., K^+ , Ca^{2+} , and Mg^{2+} , on the photocatalytic performance of both BOC-001 and BOC-010 were presented in Figures 2b-d. The addition of K^+ ions enhanced the photocatalytic activity of BOC-001, whereas it negatively impacted the photocatalytic activity of BOC-010 (Figure 2b). This discrepancy highlights the distinct interactions between K^+ ions and the different crystal facets of BiOCl, resulting in distinct regulatory effects on the photocatalytic activities of both faceted BiOCl nanocrystals. Figures

2c and 2d illustrated the photocatalytic SMX degradation curves in the presence of Ca^{2+} and Mg^{2+} . In the presence of both cations, the photocatalytic degradation activity of SMX for BOC-001 and BOC-010 was significantly enhanced. For instance, with Ca^{2+} and Mg^{2+} ions present, the SMX removal efficiency of BOC-001 increased from 92.5% (in the absence of cations) to 98.8% and 98.4%, respectively, after just 20 minutes of UV irradiation. Notably, BOC-001 consistently exhibited superior photocatalytic degradation activity for SMX compared to BOC-010 under all cationic conditions.

These findings clearly demonstrate that the photocatalytic activity of BiOCl nanocrystals is significantly influenced by both their crystalline facet structure and the presence of foreign cations. The unique structure of BOC-001 appears to facilitate enhanced interactions with cations, resulting in improved degradation rates of sulfamethoxazole (SMX). This suggests that the specific arrangement of atomic facets plays a crucial role in optimizing photocatalytic performance, as the interaction with foreign cations may promote charge carrier separation and increase the availability of reactive sites on the photocatalyst surface.

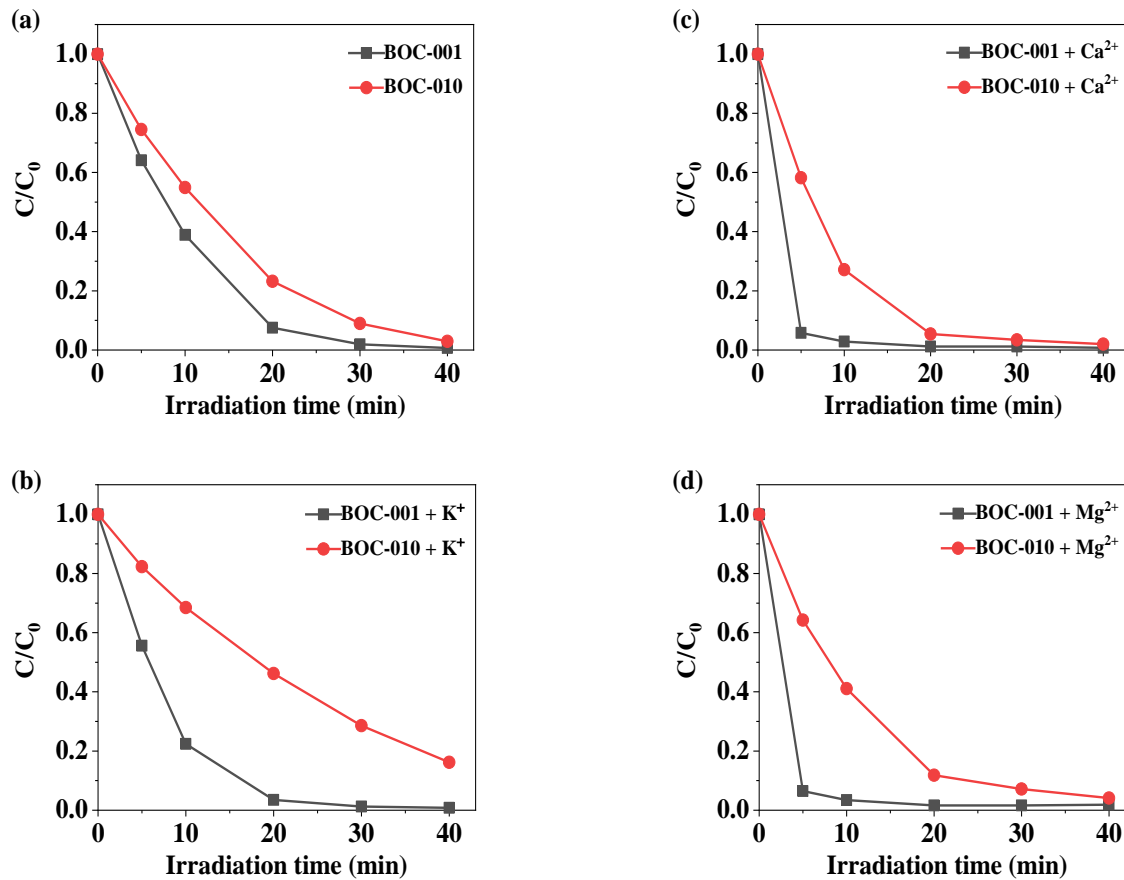


Figure 2. The photocatalytic degradation curves of SMX over faceted BiOCl nanocrystal: (a) without cations (control groups), and in the presence of (b) K^+ , (c) Ca^{2+} , and (d) Mg^{2+} .

Figure 3 presented the degradation kinetic curves of SMX over faceted BiOCl nanocrystals under varying cation conditions, which exhibits a clear adherence to pseudo-first-order kinetics.^[16] This relationship can be described by the following equation:

$$-\ln \frac{C_t}{C_0} = kt \quad (1)$$

where C_0 represents the initial concentration of SMX, C_t is the concentration at irradiation time t , k is the degradation rate constant, and t is the irradiation time. Compared to the absence of cations, the introduction of K^+ , Ca^{2+} , and Mg^{2+} ions resulted in steeper kinetic curves for the

photocatalytic degradation of SMX over both BOC-001 and BOC-010. This indicates that the presence of cations enhances the degradation rate of SMX to some extent. Particularly, BOC-001 exhibited a much steeper slope for SMX degradation across all tested conditions. This suggests that the degradation of SMX over BOC-001 occurs at a faster rate than that over BOC-010. Although the presence of these cations positively impacts the performance of BOC-001, the observed differences in degradation rates underscore the critical role of the arrangement of surface atoms and their coordination in determining the photocatalytic activity of BiOCl materials.

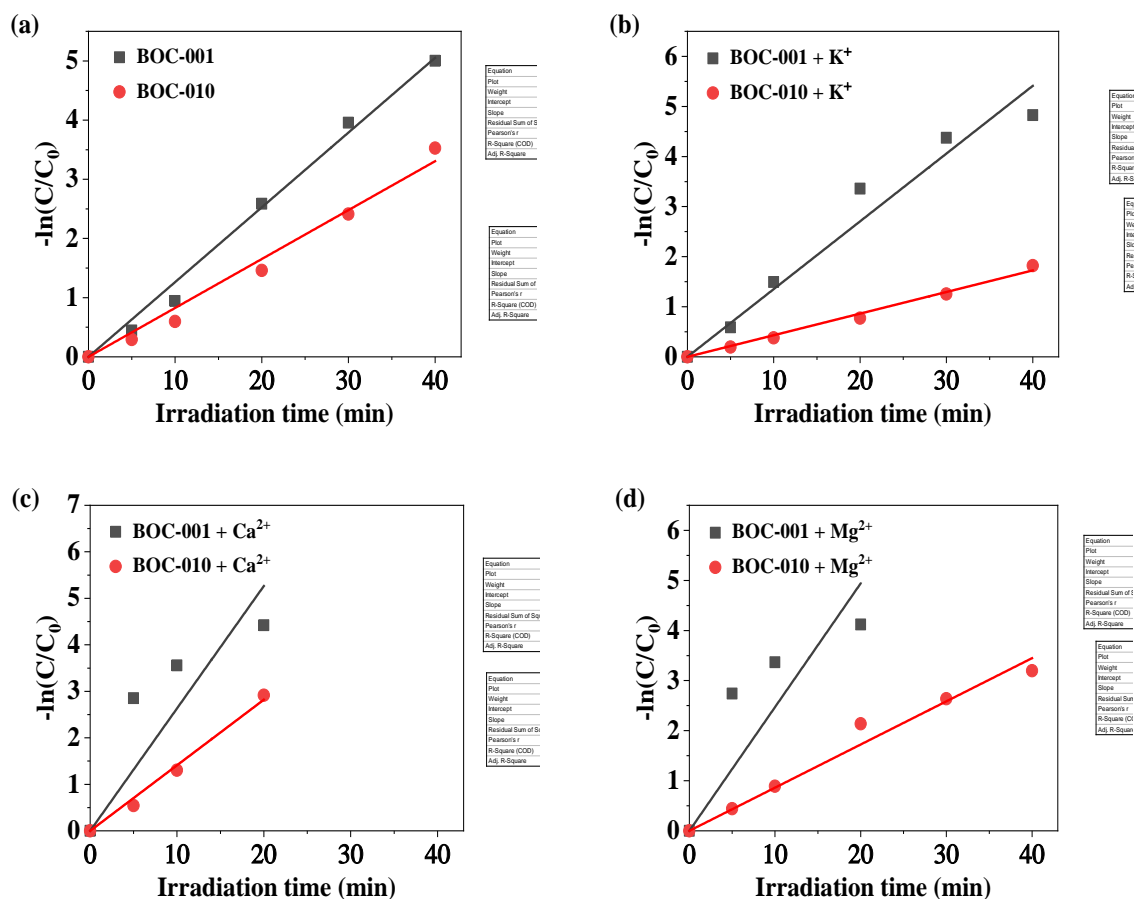


Figure 3. The degradation kinetic curves of SMX over faceted BiOCl nanocrystal (a) without cations (control groups), and in the presence of (b) K^+ , (c) Ca^{2+} , and (d) Mg^{2+} .

3.3 Effect of anions on the photocatalytic degradation of SMX

Figures 4 and 5 showed the photocatalytic degradation curves of SMX over both faceted BiOCl catalysts in the presence of various anions. The addition of different anions, such as Cl^- , NO_3^- , HCO_3^- , and SO_4^{2-} , significantly impacted the degradation efficiency of SMX over BiOCl photocatalysts. Notably, while the degradation efficiency of SMX on BOC-010 remained nearly unchanged in the presence of Cl^- , the photocatalytic activity of BOC-001 shows a substantial increase. In the presence of NO_3^- , HCO_3^- , and SO_4^{2-} ions, the photocatalytic activity over both BOC-001 and BOC-010 was significantly enhanced. For instance, without additional ions, the photodegradation efficiency of SMX over BOC-010 was 76.7% after

20 minutes of UV irradiation. With the introduction of NO_3^- , HCO_3^- , and SO_4^{2-} ions, the degradation efficiencies increased to 95.4%, 95.5%, and 96.5%, respectively.

It is noteworthy that BOC-010 exhibits a more pronounced enhancement in photocatalytic activity under NO_3^- conditions compared to BOC-001, indicating different sensitivities of the two crystal facets of BiOCl to NO_3^- . The effects of various anions on the enhancement of BiOCl photocatalytic degradation activity also differ significantly. For example, SO_4^{2-} provided a more substantial enhancement in photocatalytic activity than HCO_3^- , suggesting that the interactions between these anions and BiOCl vary depending on the specific anion present. These findings indicate that anions can enhance the effectiveness of BiOCl photocatalysts to some extent in environmental remediation applications.

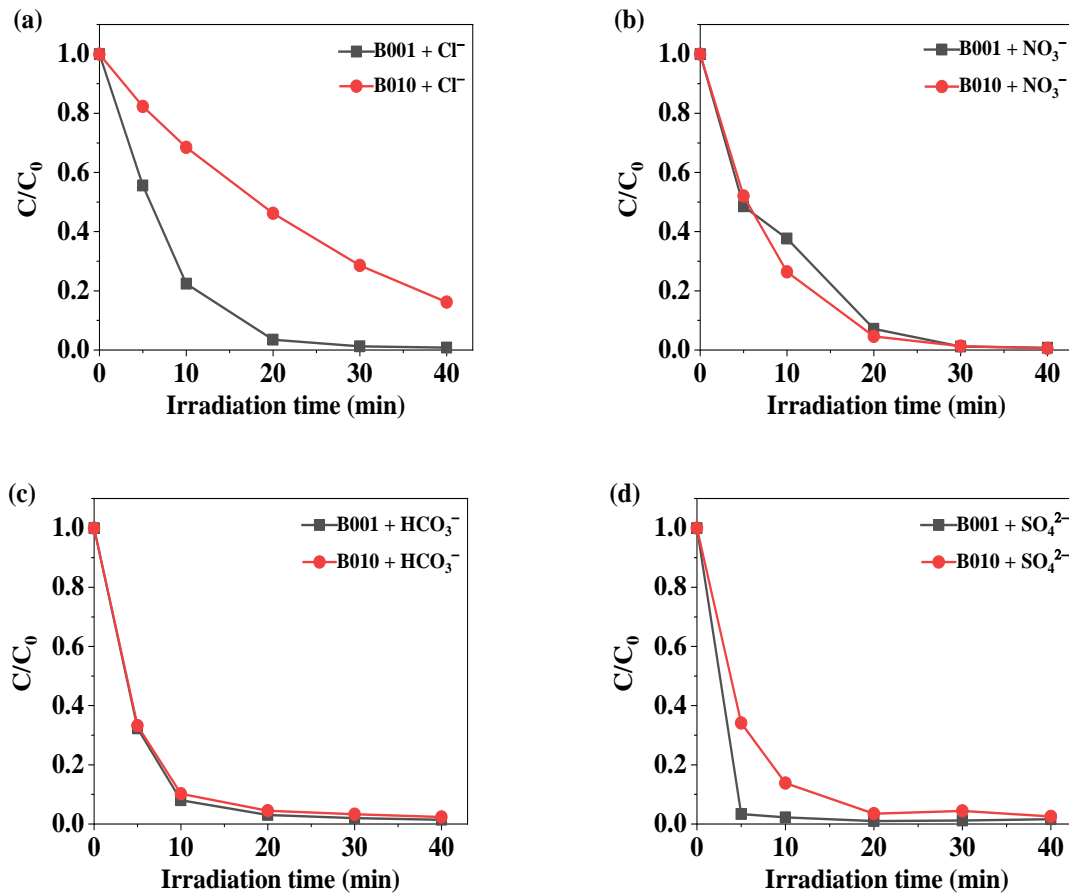
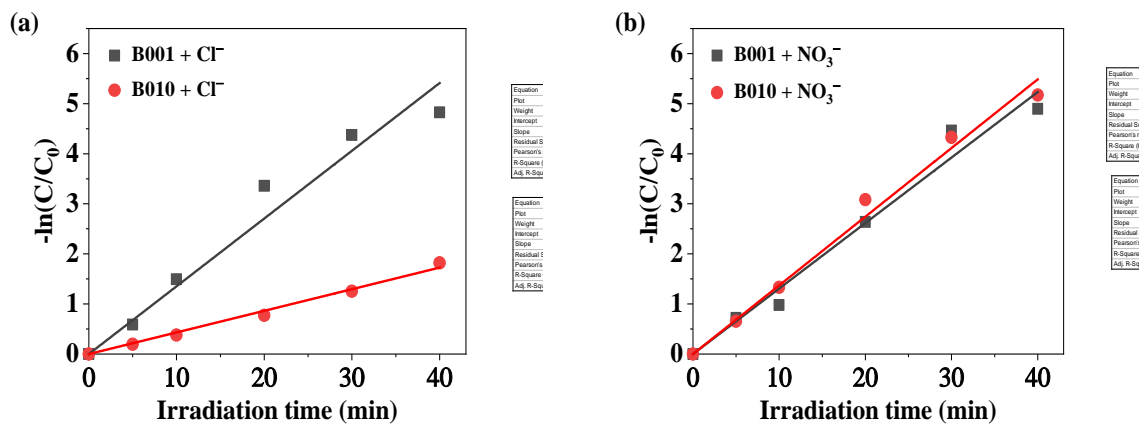


Figure 4. The photocatalytic degradation curves of SMX over faceted BiOCl nanocrystal in the presence of (a) Cl^- , (b) NO_3^- , (c) HCO_3^- , and (d) SO_4^{2-} .



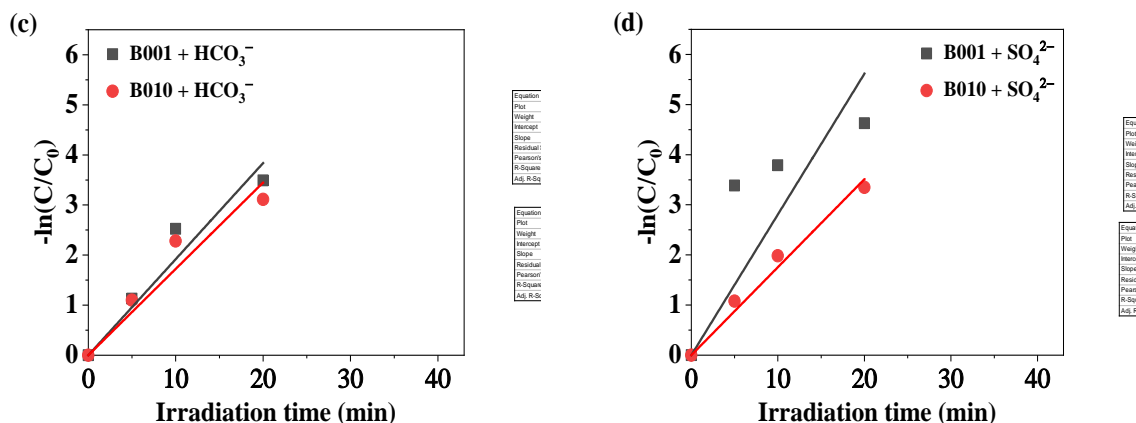


Figure 5. The degradation kinetic curves of SMX over faceted BiOCl nanocrystal in the presence of (a) Cl^- , (b) NO_3^- , (c) HCO_3^- , and (d) SO_4^{2-} .

3.4 Degradation Kinetic Rate Constant of Faceted BiOCl Nanocrystals

The degradation rate constants (k) for the faceted BiOCl nanocrystals in the presence of inorganic ions were determined using a pseudo-first-order kinetics model. As shown in Figure 6, BOC-001 demonstrated higher photocatalytic activity for SMX degradation than BOC-010 under both cationic and anionic conditions. The k for BOC-001 in the presence of Ca^{2+} , Mg^{2+} , HCO_3^- , and SO_4^{2-} were found to be 0.263 min^{-1} , 0.247 min^{-1} , 0.192 min^{-1} , and 0.281 min^{-1} , respectively, which were approximately 2.1, 2.0, 1.5, and 2.2 times of the rate constant over BOC-001 without any ions (0.126 min^{-1}). In contrast, BOC-010 photocatalyst exhibited enhanced photodegradation

performance towards SMX in the presence of Ca^{2+} , NO_3^- , HCO_3^- , and SO_4^{2-} , while the addition of K^+ and Cl^- ions negatively impacted its photocatalytic activity. It is worth noting that compared to the presence of monovalent ions, the presence of divalent ions leads to a greater increase in the degradation rate of SMX over faceted BiOCl photocatalysts. These findings suggest that the interactions between the ions and BiOCl nanocrystals are crucial for adjusting the photocatalytic performance of the faceted catalysts. Importantly, BOC-001 consistently showed a faster degradation rate for SMX than BOC-010 under all electrolyte conditions, underscoring the potential of BOC-001 as an effective photocatalyst for practical wastewater treatment applications.

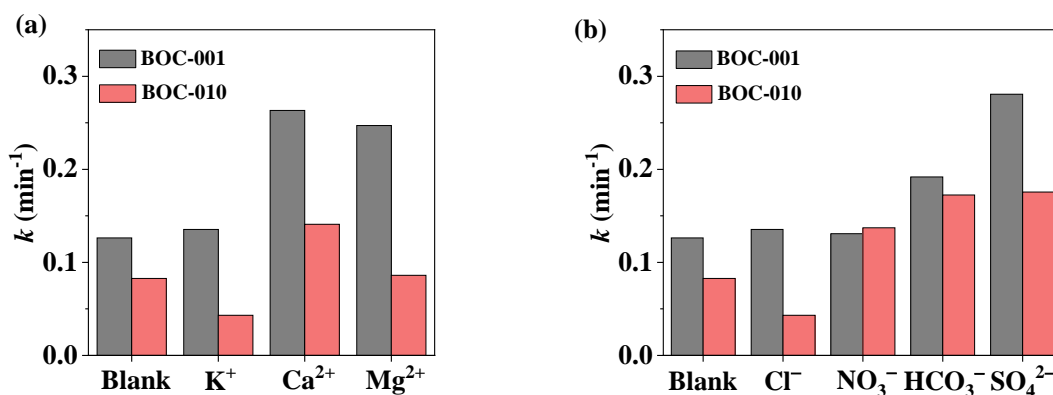


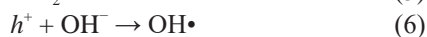
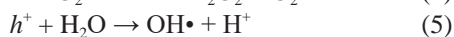
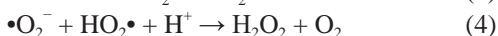
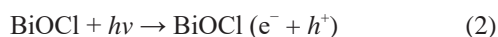
Figure 6. Comparison of the apparent reaction rate constants for photocatalytic degradation of SMX over BOC in the presence of different cations and anions.

3.5 Possible impact mechanism of inorganic ions on photocatalytic activity

The photocatalytic process in semiconductors is signifi-

cantly influenced by the electronic structure and surface properties of the photocatalyst. Although BOC-010 exhibits similar absorption edges and a smaller surface area,

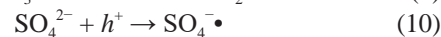
BOC-001 demonstrated markedly higher photocatalytic activity for SMX degradation. This difference in photocatalytic performance is primarily attributed to the distinct arrangement of surface atoms and their coordination of both faceted BiOCl nanocrystals. Under UV light irradiation, electron-hole pairs are generated in BiOCl. Once the charge carriers reach the surface of the catalyst, they can initiate a series of reactions with molecular oxygen and water, forming reactive oxygen species (ROS) such as superoxide radicals ($\bullet\text{O}_2^-$) and hydroxyl radicals ($\bullet\text{OH}$), which are crucial for the degradation of organic compounds:^[17]



The unique layered structure of BiOCl, characterized by alternating $[\text{Bi}_2\text{O}_2]$ layers and halogen layers, can induce the formation of internal electric fields. In BOC-001, these fields are oriented perpendicular to the nanosheets, facilitating the effective separation of photogenerated electron-hole pairs. In contrast, in BOC-010, the fields are parallel to the nanosheets, which somewhat reduces the efficiency of electron-hole pair separation.^[18] Therefore, in the BOC-001 photocatalytic system, a greater number of reactive oxygen species can be generated, leading to more pronounced catalytic activity for the degradation of SMX. Various inorganic ions present in the aqueous environment, such as Cl^- , HCO_3^- , Ca^{2+} , and Mg^{2+} , can interact with the surface of BiOCl, thus affecting the electronic structure and catalytic active sites of the catalyst. As a result, the photocatalytic activity of BiOCl nanocrystals with different exposed crystal facets is significantly influenced by the inorganic ions in the environment.

The charge characteristics of cations significantly influence the electronic distribution and band gap levels of BiOCl semiconductors. Cations with different charge properties can modify the surface potential of BiOCl, thereby affecting the recombination rate of photogenerated electrons and holes. For instance, cations such as Ca^{2+} and Mg^{2+} can form stable coordination with the active sites on BiOCl, enhancing the generation of reactive oxygen species (ROS) at the catalyst surface and improving its photocatalytic activity.^[19] Notably, BOC-001 exhibits a surface structure characterized by terminal oxygen atoms, resulting in a higher concentration of negative charges compared to BOC-010, which enhances its interaction with cations and improves the interface electron transfer and ROS generation during the photocatalytic reaction. The presence of anions also significantly affects the pho-

tocatalytic activity of BiOCl nanocrystals. The type and charge of anions are key factors influencing their adsorption on the catalytic surface. For example, divalent anions like SO_4^{2-} tend to bind more tightly to the photocatalyst surface than monovalent anions like Cl^- , leading to more substantial changes in the surface properties of BiOCl and promoting the generation of ROS.^[20] Moreover, anions have a more pronounced enhancing effect on the photocatalytic activity of BOC-010 than cations. This could be due to the open channel structure of unsaturated oxygen, bismuth, and chlorine atoms on the $\{010\}$ facet, which strengthens the interaction with anions and thus enhances their photocatalytic performance. In addition, anions such as Cl^- , HCO_3^- , and SO_4^{2-} can generate radicals like $\text{Cl}\bullet$, $\text{NO}_2\bullet$, and $\text{SO}_4\bullet^-$ under UV illumination, which also play a crucial role in the photocatalytic degradation of pollutants.^[21]



4. Conclusions

In summary, we investigated the photocatalytic activity of BiOCl catalysts with different exposed crystal facets in the presence of various inorganic ions. The observed differences in photocatalytic activity between the two BiOCl nanosheets are closely related to the interactions between inorganic ions and the surface atomic structure of the catalyst, which affect their ability to generate ROS. Regardless of the type of inorganic ion present, the photocatalytic activity of BOC-001 in degrading pollutants under UV light is consistently higher than that of BOC-010. These findings would be helpful for optimizing the design of photocatalytic systems, which has significant implications for sustainable water treatment strategies.

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