Unique Surface Chemical Species on Indium Doped TiO₂ and Their Effect on the Visible Light Photocatalytic Activity

Enjun Wang,† Wensheng Yang,‡ and Yaan Cao*,†

College of Physics, Nankai University, Tianjin 300071, P.R. China, and College of Chemistry, Jilin University, Changchun 130012, P.R. China

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TiO₂ doped by different contents of indium was prepared by the sol–gel method by using titanium(IV) tetrabutoxide and indium chloride as precursors. It was revealed that a unique chemical species, O–In–Clₓ (x = 1 or 2), existed on the surface of the indium doped TiO₂. The surface state energy level attributed to the surface O–In–Clₓ species was located at 0.3 eV below the conduction band of TiO₂. The transition of electrons from the valence band of TiO₂ to the surface state energy level was responsive to visible light. The photogenerated carriers generated under visible light irradiation can be efficiently separated by the surface state energy level of the O–In–Clₓ species and the valence band of TiO₂ to contribute to the photocatalytic reaction. Consequently, the indium doped TiO₂ showed improved photocatalytic activity for photodegradation of 4-chlorophenol compared to pure TiO₂ under visible light irradiation.

1. Introduction

TiO₂ photocatalyst has attracted much attention owing to its potential application in the solution of environmental pollution during the past decades. However, TiO₂ is only sensitive to UV light due to its large band gap (3.0–3.2 eV) which results in a low efficiency to make use of solar light. Recently, great potentials have been devoted to develop TiO₂-based photocatalysts sensitive to visible light in order to make use of solar energy much efficiently. Some strategies, such as surface modification, metal or nonmetal ion doping, etc., have been widely adopted to prepare TiO₂ photocatalysts sensitive to visible light. Among these strategies, metal doped TiO₂ has been regarded as the second generation photocatalyst. Various metal doped TiO₂’s have been widely explored to improve the photocatalytic performance of TiO₂ on the degradation of various organic pollutants under visible light irradiation. For instance, Choi and co-workers reported that doping of TiO₂ with Fe³⁺, Mo⁴⁺, Ru⁴⁺, Os³⁺, Re⁵⁺, V⁴⁺, and Rh¹⁺ at the 0.1–0.5% level increased the photocatalytic activity significantly, while Co⁵⁺ and Al³⁺ doping resulted in decreased photocatalytic activity. Nagaveni et al. prepared the W³⁺, V⁵⁺, Ce⁴⁺, Zr⁴⁺, Fe³⁺, and Cu²⁺ metal ion substituted nanocrystalline anatase TiO₂ and observed most of the metal ion doped TiO₂ showed higher photocatalytic activity than the commercial TiO₂ (Degussa P25) for photodegradation of 4-nitrophenol. Our previous work proved that Sn⁴⁺ ion doped TiO₂ nanoparticle films prepared by the plasma-enhanced chemical vapor deposition method displayed a higher photocatalytic activity for photodegradation of phenol than pure TiO₂ under both UV and visible light. Furthermore, the doping of TiO₂ with a variety of other metals such as La³⁺, Pd²⁺, Cr⁴⁺, Ag⁺, and rare earth ions (Sm³⁺, Nd³⁺, Pr³⁺) has also been investigated by the researchers. It is documented that the charge carrier recombination rates and interfacial electron-transfer rates of TiO₂ can be significantly affected by the concentration, energy level, and distribution of metal ion dopants.

In recent years, TiO₂–In₂O₃ composite photocatalysts have been explored by many researchers. Shchukin et al. prepared nanocrystalline bicomponent TiO₂–In₂O₃ powders with various Ti/In ratios by the sol–gel technique using indium(III) nitrate and TiCl₄ as precursors and evaluated their activity for photodegradation of 2-chlorophenol in water. Poznyak et al. synthesized TiO₂–In₂O₃ composites by the sol–gel technique from concentrated hydrous titanium dioxide and indium hydroxide sols and studied their structures and optical and photoelectrochemical properties. González and co-workers reported the synthesis, characterization, and photocatalytic properties of In₂O₃–TiO₂ catalysts. Reddy et al. investigated the influence of In₂O₃ on the physicochemical properties of MoO₃/TiO₂ catalysts. Yang and co-workers reported that the metallic silver and semiconductor indium oxide codoped titania nanocomposites (Ag/In₂O₃–TiO₂) showed enhanced photocatalytic activity. Compared with pure TiO₂, the In₂O₃–TiO₂ composite photocatalysts showed an efficient separation of photogenerated carriers and an enhancement of photocatalytic activity under ultraviolet light irradiation. However, the visible light photocatalytic activity of TiO₂–In₂O₃ composite photocatalysts has not been well explored up until now, possibly due to the large band gap of In₂O₃ (3.7 eV).

In this work, the indium doped TiO₂ photocatalysts with response to visible light were prepared by the sol–gel technique by using Ti(OCTH₂)₄ and InCl₃ as precursors. The experiments of 4-chlorophenol photocatalytic degradation showed that the indium doped TiO₂ photocatalysts presented higher photocatalytic activity than the pure TiO₂ and nitrogen doped TiO₂ under visible light irradiation. It was revealed that a unique surface chemical species, O–In–Clₓ (x = 1 or 2), was formed on the surface of indium doped TiO₂. The surface state energy level in the band gap attributed to the O–In–Clₓ species apparently improved the response of TiO₂ to visible light and promoted the efficient separation of the photogenerated electrons and holes. The mechanism for the improved photocatalytic activity

* To whom correspondence should be addressed. Phone: +86-22-66229598. E-mail: caoyaan@yahoo.com.
† Nankai University.
‡ Jilin University.
of indium doped TiO$_2$ under visible light was discussed correspondingly.

2. Experimental Section

2.1. Catalyst Preparation. At room temperature, a certain volume of InCl$_3$ solution (0.6 mol/L) was mixed with 40 mL of anhydrous ethanol. After mixing, 12 mL of Ti(OC$_4$H$_9$)$_4$ was added dropwise to the solution under vigorous stirring. A concentrated HCl solution (12 mol/L) was added until the final pH of this mixture was about 3.5. The mixture was stirred continuously until the formation of TiO$_2$ gel. After aging at room temperature for 24 h, the as-prepared TiO$_2$ gel was dried at 100 °C, and then triturated to powder in an agate mortar. The powder was calcined at 450 °C for 2.5 h. A series of indium doped TiO$_2$ catalysts with different concentrations of indium was prepared by changing the volume of InCl$_3$ solution added in the ethanol. The catalysts were designated as TiO$_2$–In$_x$x%, where “x%” represents the nominal molar percentage content of In$^{3+}$ ions in all metal ions (In$^{3+}$ and Ti$^{4+}$) in TiO$_2$. Pure TiO$_2$ was prepared by the same procedure but by replacing the InCl$_3$ solution with deionized water. Nitrogen doped TiO$_2$ (TiO$_2$–N) was prepared by the hydrolysis of Ti(OC$_4$H$_9$)$_4$ in the presence of ammonia.$^{29}$

2.2. Characterization. The XRD patterns were acquired on a Rigaku D/max 2500 X-ray diffraction spectrometer (Cu K$\alpha$, $\lambda = 1.54056$ Å). The average crystallite size was calculated according to the Scherrer formula ($D = k\lambda/B\cos \theta$). The BET surface areas of the samples were determined by nitrogen adsorption–desorption isotherm measurement at 77 K (Micromeritics Automatic Surface Area Analyzer Gemini 2360, Shimadzu). XPS measurements were carried out with an SECA Lab 220i-XL spectrometer by using an unmonochromated Al K$\alpha$ (1486.6 eV) X-ray source. All of the spectra were calibrated to the binding energy of the adventitious C1s peak at 284.6 eV. Raman spectra were taken on a Bruker RFS 100/S Raman spectrometer by using the 1064 nm line of a Nd:YAG laser as the excitation source. The diffuse reflectance UV–visible absorption spectra were collected on a UV–visible spectrometer (UV-4100, Hitachi). The photoluminescence (PL) spectra were measured by using the 340 nm line of a nanosecond Nd:YAG laser (NL303G) as the excitation source. The experimental setup consists of a spectrometer (Spex 1702), a photomultiplier tube (PMT, Hamamatsu R943), a lock-in amplifier, and a computer for data processing. All of the measurements were carried out at room temperature (25 ± 2 °C).

2.3. Evaluation of Photocatalytic Activity. The photocatalytic degradation of 4-chlorophenol was carried out in a 70 mL glass reactor with 10 mg amounts of catalysts suspended in 4-chlorophenol solution (5 x 10$^{-3}$ mol L$^{-1}$, 40 mL, pH 5.74) under the visible light irradiation. A sunlamp (Philips HPA 400/30S, Belgium) was used as the light source, and a 400 nm filter was employed to remove ultraviolet light for obtaining of visible light. The reactor was perpendicular to the light beam and located 10 cm away from the light source. The 4-chlorophenol solution was continuously bubbled by O$_2$ gas at a flux of 5 mL min$^{-1}$ under magnetic stirring at 25 ± 2 °C. Every 2 h, the residual concentration of 4-chlorophenol was measured by a UV–visible spectrometer (UV-1061PC, SHIMADZU) by using 4-aminoantipyrine as the chromogenic reagent. Prior to photocatalytic reactions, the suspension was magnetically stirred in the dark for 30 min, which was enough to reach the adsorption equilibrium of 4-chlorophenol. The reproducibility of the photocatalytic degradation was evaluated by repeating the experiments at least three times with different batches of the photocatalysts prepared by the same procedure. The blank experiment was performed under identical conditions. Deionized water (18.2 MΩ·cm) was used in all of the experiments, and the chemicals were all of analytical grade.

3. Results and Discussion

3.1. Structure of the Indium Doped TiO$_2$. Figure 1 shows X-ray diffraction spectra of pure TiO$_2$, TiO$_2$–In$_{3\%}$, TiO$_2$–In$_{7\%}$, TiO$_2$–In$_{10\%}$, and TiO$_2$–In$_{15\%}$. The pure TiO$_2$ (curve a) exhibited an anatase structure.$^{29}$ The position and shape of the diffraction peaks of TiO$_2$–In$_x$x% (curves b–e) were quite similar to those of TiO$_2$ (curve a), indicating after doping the crystalline phase of anatase TiO$_2$ was not changed. Except for the diffraction peaks of anatase, a weak peak at around 30.5° was observed in TiO$_2$–In$_{15\%}$ (curve e), which indicated the presence of a small amount of In$_2$O$_3$.$^{30}$ No characteristic diffraction peaks of other indium species, such as InCl$_3$, were observed in the TiO$_2$–In$_x$x% (curves b–e). The diffraction peak of crystal planes (101) was selected to estimate the crystal size of all samples by the Debye–Scherrer equation.$^{31}$ The results indicated that the crystallite size of the catalysts decreased gradually with an increase of the doping content of indium ions. At the same time, their specific surface area (BET) increased gradually, as shown in Table 1.

It is documented that there are two kinds of doping modes, interstitial and substitutional, for doped metal ions in oxides.$^{11}$ In the case of the interstitial mode, the ionic radius of dopant should be smaller than that of the lattice metal ion and the oxide lattice spacing, allowing the doping metal ions to enter into the crystal cell of the oxide. The ionic radius of the doping In$^{3+}$ ion (81 pm)$^{32}$ is larger than that of the lattice Ti$^{4+}$ ion (53 pm)$^{11}$; it is impossible for the In$^{3+}$ ions to enter into the crystal cell of TiO$_2$ through the interstitial mode. Upon doping through the substitutional mode, the doping metal ions will substitute the lattice metal ions and thus occupy the positions of the lattice metal ions in the oxide. If the ionic radius of the doping ions is larger than that of the lattice ions, the lattice parameters and cell volume of the doped oxide should be larger than those of the pure oxide. As a result, the positions of all of the diffraction peaks in XRD patterns should shift to lower diffraction angles.

It is seen that no shift of the peaks was observable in the XRD patterns of the indium doped TiO$_2$, as shown in Figure 1, and

![Figure 1](https://example.com/figure1.png)

**Figure 1.** XRD patterns of pure TiO$_2$ (a), TiO$_2$–In$_{3\%}$ (b), TiO$_2$–In$_{7\%}$ (c), TiO$_2$–In$_{10\%}$ (d), and TiO$_2$–In$_{15\%}$ (e).

**TABLE 1: Cell Parameters, Crystallite Size, and Specific Surface Area of Pure TiO$_2$ and TiO$_2$–In$_x$x% Samples**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Cell Parameters (Å)</th>
<th>Cell Volume (Å$^3$)</th>
<th>Crystallite Size (nm)</th>
<th>Specific Surface Area ($m^2g^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TiO$_2$</td>
<td>3.788, 9.516</td>
<td>136.5</td>
<td>12.6</td>
<td>68</td>
</tr>
<tr>
<td>TiO$<em>2$–In$</em>{3%}$</td>
<td>3.782, 9.524</td>
<td>136.2</td>
<td>8.8</td>
<td>76</td>
</tr>
<tr>
<td>TiO$<em>2$–In$</em>{7%}$</td>
<td>3.782, 9.535</td>
<td>136.4</td>
<td>6.2</td>
<td>112</td>
</tr>
<tr>
<td>TiO$<em>2$–In$</em>{10%}$</td>
<td>3.779, 9.518</td>
<td>135.9</td>
<td>6.0</td>
<td>110</td>
</tr>
<tr>
<td>TiO$<em>2$–In$</em>{15%}$</td>
<td>3.779, 9.535</td>
<td>136.4</td>
<td>4.9</td>
<td>114</td>
</tr>
</tbody>
</table>

![Image 370x644 to 502x749](https://example.com/image.png)
the lattice parameters and cell volume of TiO₂–Inx% samples were almost the same as those of pure TiO₂ (see Table 1). Therefore, the doping of In⁺⁺ ions into TiO₂ through the substitutional mode can also be excluded. Thus, it is reasonable to suppose that the In⁺⁺ ions are doped on the surface of TiO₂.

3.2. Surface Chemical Structure of the Doped TiO₂. XPS measurements were carried out to investigate the chemical states of all of the elements in the pure TiO₂ and TiO₂–Inx% samples. Except for C, O, Ti, In, and Cl, no other elements were detected in the samples. The peak positions of all of the elements in TiO₂ and TiO₂–Inx% samples are illustrated in Table 2. For the pure TiO₂, the peak of Cl₂p3/2 at 198.2 eV is the same as that of Cl₂p3/2 in TiCl₄ (198.2 eV), which is assigned to the Cl⁻ ions linked with Ti⁺++. Since the ionic radius of Cl⁻ ion is larger than that of O²⁻ (1.81 Å versus 1.40 Å), lattice O²⁻ in TiO₂ cannot be substituted by Cl⁻ during the reactions. Thus, it is deduced that the Cl⁻ ions should be located on the TiO₂ surface via the coordination with the unsaturated Ti⁺⁺ sites.

Figure 2a shows the In3d XPS spectra of TiO₂–Inx% samples. The peaks at 444.8–445.3 eV and 452.4–452.9 eV are assigned to In 3d₅/₂ and In 3d₃/₂, respectively. The binding energies of In 3d₅/₂ (444.8–445.3 eV) in TiO₂–Inx% samples are located between those of In 3d₅/₂ in InCl₃ (444.6 eV) and In 3d₅/₂ in InCl₄ (446.0 eV). In the Cl 2p XPS spectra of TiO₂–Inx% (see Figure 2b), the binding energies of Cl₂p3/2 are identified to be 198.5–198.6 eV, which are located between those of Cl 2p3/2 in TiCl₄ (198.2 eV) and Cl 2p3/2 in InCl₃ (199.1 eV). These results suggest that the In⁺⁺ ions link with the unsaturated oxygen and Cl⁻ ions, respectively, thus meaning the formation of O⁻–In⁻Cl⁻ (x = 1 or 2) structure on the surface of TiO₂–Inx%. A part of the Cl⁻ ions may also link with the unsaturated Ti to form the O⁻–Ti–Cl structure. Raman spectra were recorded to further investigate the formation of the surface species (Figure 3). All of the samples show characteristic bands at 142, 195, 395, 515, and 637 cm⁻¹, which are assigned to the E₆, B₁₈₉, A₁₈₉, B₂₉, and E₉ vibrational modes of TiO₂, respectively, indicating the presence of the anatase phase in all of these samples. Except these peaks, some additional peaks around 300, 470, and 618 cm⁻¹ were observed for the TiO₂–In7% and TiO₂–In10% samples. Gómez et al. have reported that three peaks at 288, 467, and 608 cm⁻¹ were observed in the Raman spectrum of In₂O₃. Therefore, we ascribed these peaks to the vibrational modes of In–O bonds. Furthermore, in comparison to the Raman peak of InCl₃ at 280 cm⁻¹, the peaks around 275 cm⁻¹ are supposed to be related to the In–Cl bond. The Raman spectra suggested the existence of both In–O and In–Cl bonds as the surface chemical species of the In doped TiO₂, which is consistent with the results of XPS that O⁻–In⁻Cl⁻ was formed on the surface of the doped TiO₂.

On the basis of the above experimental results and discussion, we proposed a mechanism for the formation of surface species in the doped TiO₂ (Scheme 1). In the sol–gel process, the TiO₂ could rapidly precipitate at a pH of 3.5 and InCl₃ could only be partially hydrolyzed. Thus, some In species, such as InCl₃, InCl₃OH, and InCl(OH)₂, may be absorbed on the surface of TiO₂. It is expected that these species would link with the unsaturated oxygen on the surface of TiO₂ to form the O⁻–In⁻Cl⁻ structure and some Cl ions would link with the unsaturated Ti to form the O⁻–Ti–Cl structure during the subsequent calcination process.

Pure TiO₂ shows Ti2p peaks assigned to Ti 2p₃/₂ and Ti 2p₁/₂ at 458.3 and 464.0 eV (Figure 4) and an O1s peak assigned to lattice O²⁻ at 529.5 eV (Figure 5). In the case of TiO₂–Inx% samples, all of the peaks of Ti2p and O1s of TiO₂ present higher binding energies after the doping of In⁺⁺, which is consistent with the formation of O⁻–In⁻Cl⁻ structure on the TiO₂ surface after the doping. The Pauling electronegativity of In⁺⁺ ion (1.7) in the Ti–O–In–Cl⁻ structure is larger than that of the Ti⁺⁺ ion (1.5) in the Ti–O–Ti structure, which will induce the possible electron transfer from Ti⁺⁺ and O²⁻ to In⁺⁺ in the Ti–O–In–Cl⁻ structure. Such a charge transfer will make Ti⁺⁺ and O²⁻ be poorer in electrons and thus result in an increase in the binding energies of Ti 2p and O 1s core electrons. It is noted that, in Figure 5, the O 1s signals present shoulders located at the side of higher binding energy, which are assigned to the OH species on the surface.
TiO

indicating after the doping of indium ions the band gaps of (e). The inset gives the difference DRS spectra of TiO

at 350 nm attributed to the transition of band being doped by indium.

TiO

(b), TiO

were obtained by subtracting the absorbance spectra of pure TiO

have almost no shift compared with pure TiO

band of TiO

The band threshold at 400 nm corresponds to the electronic transition from the valence band to the conduction band, the inset in Figure 6. It is deduced that the remarkable absorption peak at 400 nm was ascribed to the electronic transition from the valence band of TiO

to the surface state energy level of TiO

atoms into + ions. This observation further suggested that the surface state energy level attributed to the O−In−Cl species on the surface of TiO

are responsible for the visible light absorption of the doped TiO

the surface of pure TiO

was treated with concentrated HCl to replace the surface OH groups of TiO

with Cl−, which resulted in the formation of O−Ti−Cl species. However, there is no difference between the absorption spectra of pure TiO

and the treated TiO

samples (see Figure S1 in the Supporting Information). Thus, it is reasonable to attribute the visible light absorption to O−In−Cl species on the surface of the doped TiO

The surface state energy level contributed by the O−In−Cl species is likely to locate at about 0.3 eV below the conduction band of TiO

which is further supported by the laser photoluminescence spectral measurements in the following part. The absorption peak at 400–800 nm was ascribed to the electronic transition from the valence band of TiO

to the surface state energy level of the O−In−Cl species.

3.4. Photoluminescence Spectra. Figure 7 shows the PL emission spectra of pure TiO

and TiO

samples. Two peaks around 480 and 525 nm are observed for the pure TiO

(Figure 7a), attributed to the transition from the oxygen vacancies with two trapped electrons and one trapped electron to the valence band of TiO

respectively. The energy levels related to the two kinds of the oxygen vacancies are located at 0.51 and 0.82 eV below the conduction band of TiO

respectively. Due to the existence of the energy levels of oxygen vacancies, first the photogenerated electrons in the conduction band should reach the oxygen vacancies through a nonirradiative process, and then recombine with the photogenerated holes in the valence band accompanied by the emission of fluorescence. It can be seen that the emission intensity of TiO

is weakened significantly in TiO

samples. In order to make clear what species are responsible for the visible light absorption of the doped TiO

hydroxyl groups on the TiO

surface increased greatly after being doped by indium.

3.3. Response of the Doped TiO

to Visible Light. The diffuse reflectance UV−visible absorption spectra of pure TiO

and TiO

samples are given in Figure 6. The pure TiO

(curve a) shows a strong peak at 350 nm, which is attributed to the electronic transition from the valence band to the conduction band of TiO

The band threshold at 400 nm corresponds to a band gap of 3.1 eV. For TiO

samples, absorption peaks at 350 nm attributed to the transition of band−band of TiO

have almost no shift compared with pure TiO

ing the inset in Figure 6. The position of the absorption maximum was around 440 nm, corresponding to an energy level gap of about 2.8 eV. Moreover, the intensity of absorption in the visible region increased with the increased content of In

ions doped in TiO

curves b−e, the inset in Figure 6). Thus, it is deduced that the remarkable absorption at 400–800 nm is related to the doped In

ions. According to the analyses of XPS, after doping In

ions into TiO

the O−In−Cl and O−Ti−Cl species existed on the surface of TiO

−In% samples. Thus, the emission intensity of TiO

−In% decreased greatly after being doped by In

ions.
3.5. Photocatalytic Activity. The photocatalytic activity of pure TiO₂, nitrogen doped TiO₂, and TiO₂–Inₓ% samples, and the experimental results of the photocatalytic activity of pure TiO₂, nitrogen doped TiO₂, and TiO₂–Inₓ% samples are summarized in Table 3. The apparent rate constant and the specific photocatalytic activity of TiO₂ were calculated from the linear fitting of ln(c/kc) versus reaction time.

<table>
<thead>
<tr>
<th>Sample</th>
<th>4-CP degraded, (%)</th>
<th>k² (min⁻¹)</th>
<th>t½ (min)</th>
<th>Specific photocatalytic activity (mol g⁻¹ h⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure TiO₂</td>
<td>5.2 ± 0.2</td>
<td>(1.13 ± 0.02) × 10⁻⁴</td>
<td>6.10 ± 0.07</td>
<td>10⁻¹</td>
</tr>
<tr>
<td>TiO₂–N</td>
<td>8.2 ± 0.5</td>
<td>(1.72 ± 0.04) × 10⁻⁴</td>
<td>3.92 ± 0.11</td>
<td>10⁻¹</td>
</tr>
<tr>
<td>TiO₂–In3%</td>
<td>18.0 ± 2.1</td>
<td>(3.90 ± 0.54) × 10⁻⁴</td>
<td>1.79 ± 0.23</td>
<td>10⁻¹</td>
</tr>
<tr>
<td>TiO₂–In7%</td>
<td>38.8 ± 3.7</td>
<td>(1.02 ± 0.14) × 10⁻³</td>
<td>6.47 ± 0.84</td>
<td>10²</td>
</tr>
<tr>
<td>TiO₂–In10%</td>
<td>37.8 ± 3.5</td>
<td>(9.69 ± 1.32) × 10⁻²</td>
<td>7.12 ± 0.88</td>
<td>10²</td>
</tr>
<tr>
<td>TiO₂–In15%</td>
<td>31.6 ± 2.9</td>
<td>(7.88 ± 0.97) × 10⁻⁴</td>
<td>8.78 ± 0.78</td>
<td>10²</td>
</tr>
</tbody>
</table>

*After reaction for 8 h. *Apparent rate constant deduced from the linear fitting of ln(c/kc) versus reaction time.

4. Conclusion

A new kind of TiO₂-based visible light photocatalysts with a surface chemical structure of O–In–Cl, was successfully prepared by doping of indium ions. The photocatalysts present higher photocatalytic activity than pure TiO₂ and nitrogen doped TiO₂ under visible light irradiation. The surface energy level located at 0.3 eV below the conduction band attributed to the formation of In₂O₃ at the higher content of indium, as indicated in Figure 1, which will cover the surface of the catalyst and retard the charge transfer from the indium doped catalysts to O₂ or 4-chlorophenol molecules.

References and Notes

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