Abnormal SPR-Mediated Photocatalytic Enhancement of Ag Nanocubes Covered by AgCl Ultra-thin Layer

Yanjiao Li1 · Tiago V. Alves2 · Xiaoyun Liu3 · Mai Li1 · Chunrui Wang1 · Jiale Wang1,4

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Abstract
It was theoretically predicted that electric field intensities generated by surface plasmon resonance (SPR) around plasmonic metallic nanostructures could be reduced after covered by wide bandgap semiconductor, thus worsening the SPR-mediated photocatalytic activities. However, we found that the Ag nanocubes (Ag NCs) covered by AgCl demonstrated an abnormal SPR-mediated catalytic enhancement. Herein the PATP-to-DMAB oxidization was used as a model reaction to explore the photocatalytic conversions. It was observed that the Ag NCs covered by AgCl ultra-thin layer (AgCl@Ag NCs) presented better photocatalytic activities than those of individual Ag NCs under 633-nm excitation. To reveal the mechanism behind the superior activities of AgCl@Ag NCs, the PATP-to-DMAB conversions were also carried out in ambient argon (Ar), in which AgCl@Ag NCs demonstrated significantly higher conversions. These results could be interpreted based on the separation of electron–hole pairs in AgCl through SPR effect of Ag, where the electrons in the valence band of AgCl were excited to the empty states of Ag, leaving holes in the semiconductor and resulting in the oxidation of PATP to DMAB. Thus, our findings may provide a novel way to enhance SPR-mediated photocatalytic activities on plasmonic metallic nanostructures by the cover of an ultra-thin layer of wide bandgap semiconductor.

Keywords Ag · AgCl · Surface plasmon resonance · Photocatalysis

Introduction
Recently, surface plasmon resonance (SPR) has showed great application in various areas, e.g., sensor, photovoltaics, photocatalysis etc. [1–3]. Since SPR can be triggered by visible light [4, 5], which constitutes ~43% of the solar energy [6, 7], thus SPR-mediated photocatalysis is treated as a promising strategy to drive the green catalytic processes [8, 9]. Silver (Ag), as one of the extensively studied plasmonic metallic materials, displays various characteristics in visible light range relative to its nanostructures of different shapes, e.g., sphere, wire, bipyramid, cube etc. [10–14].

In nanocatalysis, metal nanoparticles (NPs) can be deposited on semiconductor supports to avoid aggregation or/and facilitate recovery [15]. Moreover, if there exist metal-support interactions, the catalytic performance of metal NPs can be enhanced [16, 17]. Recently, AgCl is typically used as support for Ag NPs deposition, and it can raise the visible-light responsive photocatalytic performances by the separation of carriers [18–21]. However, generally it is difficult to control the morphology of Ag nanostructures deposited on AgCl. Thus, we considered an opposite way to cover the Ag nanostructures of certain morphology by AgCl. Since near-field coupling of Ag nanocubes (Ag NCs) exhibited nearly twice the decay length relative to that of nanospheres [22, 23], thus Ag NCs were selected as model nanostructure. The SPR-mediated photocatalytic activities of Ag NCs were compared with those covered by AgCl.

Since the plasmonic photocatalytic activity is directly proportional to the square value of local electric field (E-field)
intensity generated upon SPR [24–27], theoretical E-field contour on an individual Ag NC of 83 nm in edge length was carried out by discrete dipole approximation (DDA) method [28] employing 633 nm as the excitation wavelength (Fig. S1a). Meanwhile, similar analysis was also done on an Ag NC covered by AgCl (AgCl@Ag NC) for comparison, where an AgCl@Ag NC with Ag core of 83 nm in edge length and 2 nm-thick AgCl layer covered was used (Fig. S1b). Figure S1c showed that the E-field intensities were elevated at the corners of the Ag NC. The maximum $|E|^2 / |E_0|^2$ value was 42.57, where $E$ and $E_0$ correspond to the local and incident light E-field intensities, respectively. Meanwhile, the maximum $|E|^2 / |E_0|^2$ value of AgCl@Ag NC was 36.94 as presented in Fig. S1d. It seemed that AgCl@Ag NCs would display inferior SPR-mediated photocatalytic activities relative to those of Ag NCs. However, in this paper we demonstrated that the plasmonic photocatalytic activities of AgCl@Ag NCs were superior relative to those of Ag NCs, where the p-aminothiophenol to p,p’-dimercaptoazobenzene (PATP-to-DMAB) oxidation was chosen as a model reaction [29].

Together with the PATP-to-DMAB conversions carried out in ambient argon (Ar), the mechanism could be explained based on the separation of electron–hole pairs in AgCl through SPR effect of Ag NCs, where the electrons in the valence band (VB) of AgCl were excited to the empty states of Ag, leaving holes in the semiconductor and enhancing the oxidation of PATP. Thus, our work may provide a facile avenue to enhance SPR-mediated photocatalytic activities on plasmonic metallic nanostructures of certain shapes by covering an ultra-thin layer of wide bandgap semiconductor.

**Results and Discussion**

Firstly, Ag NCs were synthesized by hydrothermal method as previously reported [30]. Figure 1a presents the SEM image of Ag NCs, which displayed good cube-like morphology with relatively sharp corners and edges. The size was relatively monodisperse with ~83 nm in edge length. The TEM image shows that the surface of Ag NC was smooth...
The HRTEM image depicted in Fig. 1b displays the lattice spacing of 2.0 Å. This value was assigned to Ag(200) interplanar distance and coincided with the SAED results in Fig. S2b [31]. Figure S2c presents the enlarged view of the area enclosed by dashed lines in Fig. S2b, where all the points display a good dot-like shape.

Then AgCl thin layer was covered on the surface of Ag NCs to form AgCl@Ag NCs sample. Figure 1c presents the SEM image of AgCl@Ag sample, which demonstrated a similar cube-like morphology as that of Ag NCs. However, the average size of AgCl@Ag NCs increased to ~87 nm, and the core–shell structure can be clearly observed in Fig. S2d, where a ring of different contrast was located outside the Ag NC. Moreover, the core–shell structure was more obvious in the HRTEM image (Fig. 1d). The central part exhibited a lattice spacing of 2.4 Å, whose value was coincident with Ag (111) interplanar distance and coincided with the SAED results in Fig. S2e [32]. Meanwhile, the ~2 nm-thick layer outside presented a lattice spacing of 2.8 Å, which is smaller than that of AgCl (111) interplanar distance (3.2 Å) [33], revealing the epitaxial growth of AgCl on Ag NCs. Furthermore, the enlarged view of the area enclosed by dashed lines in Fig. S2e shows that all the points consisted of two dots (Fig. S2f), and it indicates that the SAED of AgCl@Ag NC was the superposition of two different patterns [34]. Both Ag and AgCl have the same body-centered cubic (BCC) lattice structure, and their lattice constants are 4.08 Å (Ag) and 5.54 Å (AgCl), respectively [34–37]. The ultra-thin AgCl layer grown on Ag can keep the strain energy unreleased [38], and thus, the sharp interface between AgCl and Ag can prefer the charge transfer between them [19].

Figure 2a presents the XPS survey spectra of Ag (top) and AgCl@Ag NCs (below), respectively. With exposure under UV irradiation for ~10 min, intensities of surface C contamination on Ag and AgCl@Ag NCs samples demonstrated ~5 times and ~4 times less than those of Ag 3d.
respectively. Figure 2b–d correspond to the Ag 3d and Cl 2p core-level spectra and the binding energy (BE) values were in Table S1.

In Fig. 2b, the Ag 3d core-level spectrum of Ag NCs sample presented two peaks with BE at 367.83 and 373.83 eV. These two peaks were attributed to Ag 3d5/2 and Ag 3d3/2 of Ag, respectively [39]. The Ag core-level spectrum of the AgCl@Ag NCs samples shown in Fig. 2c demonstrated 2 components for both Ag 3d5/2 and Ag 3d3/2 bands, respectively. The peaks at 367.83 and 373.83 eV corresponded to Ag0 [39], while the peaks at 367.23 and 373.23 eV were ascribed to Ag+ of AgCl [40]. The appearance of Ag+ peaks indicates the formation of AgCl as reported previously [41, 42]. Meanwhile, the detection of Cl 2p core-level spectra in Fig. 2d provided another proof of the formation of AgCl. Two peaks with BE at 197.64 and 199.28 eV corresponded to Cl 2p3/2 and Cl 2p1/2, respectively [43, 44].

Then we focus on the plasmonic photocatalytic activities of Ag and AgCl@Ag NCs samples to investigate the influences on the cover of AgCl thin layer. The PATP-to-DMAB conversions were chosen as a model reaction [45, 46], because Raman peaks of PATP and DMAB can be easily monitored and are at different positions.

Figure 3a, b display the SERS spectra carried out in ambient air on Ag and AgCl@Ag NCs samples, respectively. The 633-nm excitation was employed with 6 s exposure time. All the peaks observed correspond to A1 modes of PATP and A2 modes of DMAB as reported previously, and the information has been directly marked on the figure [47]. All spectra were normalized with respect to the peak at 1081 cm−1, and then the DMAB/(PATP + DMAB) intensity ratios can be directly obtained from the intensities of the most obvious peak corresponding to DMAB at 1433 cm−1 [48–51].

For Ag NCs sample (Fig. 3a), a weaker DMAB peak at 1433 cm−1 could be detected at 0.045 mW. With the power increased, this peak was more and more obviously detected. This photocatalytic performance corresponded to the SPR effect of Ag NCs as previously reported [52, 53]. For AgCl@Ag NCs sample (Fig. 3b), the DMAB peak at 1433 cm−1 presented a similar increasing tendency as those of Ag NCs sample with the elevation of laser power. However, the DMAB peak intensities of AgCl@Ag NCs sample were always higher than those of Ag NCs sample under irradiation of same laser power as shown in Fig. 4, indicating superior SPR-mediated photocatalytic activities on AgCl@Ag NCs sample.

The free electrons in plasmonic metallic nanostructures are collectively oscillated induced by the electromagnetic field of incident light upon SPR excitation [54]. At a certain time, the free electrons will aggregate at one side in a nanostructure, and meanwhile, the empty states of electrons (holes) are left on the other side in the same nanostructure [55–57]. It has been proved that the holes are exclusive for the oxidation of PATP [58–60]. However, the electrons and holes generated upon SPR excitation can be quenched very quickly in a recombination process [61]. Therefore, the O2 from air around samples can functionalized as an electron...
The capturer to consume part of the free electrons, leaving holes for the oxidation of PATP [61].

Since the bandgap of AgCl is 3.3 eV [62], thus 633-nm irradiation cannot excite electrons from its valence band (VB) to conduction band (CB) directly. However, when AgCl forms Schottky contact with Ag as in Fig. 5a, electrons on the valence band (VB) of AgCl can be excited to the empty states of Ag under irradiation of 633-nm excitation. Moreover, the work function of Ag is 4.3 eV, and the work function of AgCl is 4.7 eV [63, 64]. Thus, the energy bands of AgCl bend towards smaller energy values when Schottky contact is formed between AgCl and Ag [34], which will inhibit the transfer of holes to Ag. Since the PATP-to-DMAB conversions are normally limited by the quantity of holes [58], the cover of AgCl on Ag NC leads to the separation of electron–hole pairs in AgCl through SPR effect of Ag NC, where the electrons in the valence band of AgCl are excited to the empty states of Ag, leaving holes in the semiconductor and enhancing the oxidation of PATP. To confirm this mechanism, the PATP-to-DMAB conversions were then investigated in ambient Ar (Fig. 3c, d).

As shown in Fig. 4, for the Ag NCs sample the DMAB peak intensities at 1433 cm⁻¹ carried out in ambient Ar were always lower than those done in ambient air under irradiation of same laser power, which is consistent with the results reported previously that the PATP-to-DMAB conversions on plasmonic metallic nanostructures were quite lower in ambient inert gas [61]. However, for the AgCl@Ag NCs sample the intensities of the same DMAB peak measured in ambient Ar were always higher than those done in ambient air. Since the AgCl layer was ultra-thin, thus some free electrons in Ag might penetrate the AgCl layer through tunneling effect upon SPR excitation [65], which could be captured by the adsorbed O₂ molecules from air to generate activated O₂ [60]. Subsequently, the recombination of activated O₂ with part of the holes in AgCl could reduce the PATP-to-DMAB conversions carried out in ambient air, and thus, the PATP-to-DMAB conversions displayed an enhancement in ambient Ar. Moreover, the SERS was carried out on Ag NCs covered by 5 nm AgCl (Fig. S3) both in ambient air and in ambient Ar. As presented in Fig. S4, it was observed that the PATP-to-DMAB conversions were inferior on Ag NCs covered by 5 nm AgCl relative to Ag NCs covered by 2 nm AgCl. Since more holes will be recombined after they pass a longer route inside AgCl [63], the mechanism in Fig. 5 was further confirmed by the results that the Ag@AgCl NCs with thicker AgCl displayed a worse photocatalytic performance.

![Figure 4](image_url) 1433:1081 cm⁻¹ DMAB:(PATP + DMAB) intensity ratios as a function of the laser power obtained from Fig. 3

![Figure 5](image_url) a Schematic for the energy-level diagrams and charge transfer pathways between Ag and AgCl under 633-nm irradiation. b Schematic illustration for the PATP-to-DMAB oxidation on Ag@AgCl NC under 633-nm irradiation
It was noteworthy that the morphology of Ag and AgCl@Ag NCs did not change after photocatalytic reaction as shown in Fig. S5a and b, indicating the stability of their structures under light irradiation.

Conclusions

In summary, we herein reported a facile avenue to elevate SPR-mediated photocatalytic activities of Ag NCs by covering an ultra-thin layer of AgCl. We found that the separation of electron–hole pairs in AgCl through SPR effect of Ag could result in the enhancement of SPR-mediated photocatalytic performances, where electrons in the valence band of AgCl were excited to the empty states of Ag, leaving holes in the semiconductor and resulting in the oxidation of PATP induced by Au NPs and Au@Pd NPs supported on graphene. This work may provide a novel way to elevate SPR-mediated photocatalytic activities on plasmonic metallic nanostructures by the cover of an ultra-thin layer of wide bandgap semiconductor.

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Author Contribution Y.L. fabricated the samples and wrote the main manuscript. T.A. did the DDA simulation. X.L. helped with the Raman measurement. M.L. and C.W. analyzed data and revised the main manuscript. J.W. conceptualized and supervised the research.

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Data Availability The data in this study are available from the corresponding author on reasonable request.

Declarations

Competing Interests The authors declare no competing interests.

Ethics Approval Not applicable.

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Consent for Publication Not applicable.

Conflict of Interest The authors declare no competing interests.

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