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MINI REVIEW

Ag/AgX nanostructures serving as antibacterial agents: achievements and challenges

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Abstract Bacterial infections, especially the frequently emerging "superbugs", seriously affect the quality of human life and even threaten human health. As the emerging antimicrobial agents that effectively eradicate pathogens, nanomaterials have been widely explored due to their effectiveness against wide-spectrum bacteria and "superbugs". Of them, Ag/AgX nanostructures (X representing Cl, Br or I) have emerged as an excellent antibacterial agent because of their excellent photocatalytic performance in inactivating pathogens under light irradiation, which provides a new opportunity for the development of high-efficient visible-light driven photocatalytic sterilization. To date, Ag/AgX nanostructures have been widely employed in antibacterial associated fields because they are efficient in producing reactive oxygen species (ROS) and reactive chlorine species (RCS) under visible light irradiation. In this review, we summarized the recent progress of Ag/AgX nanostructures as plasmonic photocatalysts in the antibacterial field, focusing on the antibacterial effects and mechanisms of Ag/AgX nanostructures, as well as their potent applications. Finally, the challenges and prospects of Ag/AgX nanostructures acting as active antibacterial agents were discussed.

Keywords Ag/AgX; Photocatalytic; Antibacterial; Nanostructure; Nanoengineering

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1 Introduction

Bacterial contamination has been recognized as one of the great challenges to human health [1]. Especially, multi-drug resistance is a growing issue in the treatment of infectious diseases, and the abuse of broad-spectrum antibiotics has prompted antibiotic resistance for many pathogens [2–4]. At present, some semiconductors with excellent photocatalytic activity, such as ZnO, MoS_2 and TiO_2, have been explored as promising antimicrobial agents to kill bacteria [5–7]. The antimicrobial actions of semiconductor-based photocatalysts are attributed to the production of reactive oxygen species (ROS) under light irradiation, which induce damage to cellular and extracellular components, including DNA, proteins and lipids. Of the various semiconductors that effectively inactivate pathogens, TiO_2-based photocatalysts are considered as "green" materials for widespread biomedical applications, due to their high activity, long-term stability, low cost, and nontoxicity [8]. Unfortunately, TiO_2-based photocatalyst just can be activated by ultraviolet (UV) light, which is only about 4% of the sunlight spectrum [9, 10]. Therefore, in order to improve the utilization of solar energy, the development of new and more efficient visible light-driven (VLD) photocatalytic antimicrobial agents has become an attractive topic.

Silver halides (AgX, where X represents Cl, Br or I) are generally employed as important inorganic photosensitive materials, which have high sensitivity in the visible light (VL) region and are widely used in photographic imaging technology [11]. However, AgX suffers from photosensitivity and instability under light irradiation. Accordingly, Huang's group first prepared an Ag/AgCl plasma photocatalyst with high photocatalytic activity and stability by combing Ag with AgCl [12]. Owing to the strong surface plasmon resonance

(SPR) effect of Ag nanoparticles (AgNPs), the light absorption range of Ag/AgCl shifted from UV to visible region, and the inhibition of electrons and holes of the composite can be rose, endowing Ag/AgCl high visible light catalytic activity. Subsequently, their group also designed a series of Ag/AgX (X = Cl and Br) plasma photocatalysts, which have good photocatalytic activities for the removal of organic pollutants, bacterial inactivation, and the reduction of toxic heavy metal ions under VL irradiation, as shown in Fig. 1. Since then, Ag/AgX nanostructures were gradually used to inhibit and kill pathogens under VL irradiation [13].

To date, numerous studies have explored the antibacterial action of Ag/AgX nanostructures. Typically, the antibacterial mechanisms of Ag/AgX nanostructures can be classified into three types: (i) Ag^+ damages the permeability of bacterial cell membranes and denatures bacterial proteins [14]. (ii) Ag/AgX nanostructures produce active species under VL irradiation, such as reactive oxygen species (ROS) and reactive chlorine species (RCS), which damage cell membranes and cell walls to destroy the defense system of bacteria [15, 16]. (iii) Ag/AgX nanostructures are able to enhance the absorption of VL and provide new opportunities to develop VLD photocatalysts [17]. Therefore, Ag/AgX nanostructures can be coupled with other materials to achieve highly effective antibacterial function via a synergistic antibacterial mechanism [1, 18–21]. Typically, Ag/AgX can also be dispersed on carbon composite and semiconductor composite to improve the stability and antibacterial activity for potential applications, with Ag/AgCl/rGO [15] and Ag/AgBr/TiO_2 [22] being two typical examples.

Several reviews have reported on the past progress of some photocatalysts as antibacterial agents for bacterial decontamination. For instance, Wang et al. [23] have summarized the recent progress and challenges of photocatalytic nanomaterials for solar-driven bacterial inactivation. You et al. [6] reviewed the visible light-active photocatalysts via a variety of strategies for water disinfection. However, as far as we know, no review has been reported on Ag/AgX nanostructures, which function as an antibacterial agent. In this review, we summarize the recent advances on Ag/AgX nanostructures and their nanoengineering for antibacterial use, focusing on the antibacterial mechanism, structural engineering, and some factors affecting antibacterial ability, as well as their antibacterial application. Finally, we provide valuable insights into the future development of Ag/AgX nanostructures in the antibacterial fields.

2 Antibacterial mechanisms of Ag/AgX nanostructures

Antibacterial actions of AgNPs and AgX against a wide range of microorganisms have been widely explored; however, the accurate bacteria-killing mechanism of Ag/AgX nanostructures is still not assumed [24]. The antibacterial mechanism of AgNPs is commonly assumed to the release of Ag^+ that react with the -SH groups of proteins, resulting in inactivating those proteins. Furthermore, the released Ag^+ possess a capability of penetrating the bacterial cell wall and membrane, which leads to death of bacterial cells. In addition to Ag^+ released mechanism, some previous literatures have reported on the plasma photocatalyst mechanisms of Ag/AgX nanostructures, via the production of reactive species under VL irradiation, such as ROS and RCS [25]. Besides, Ag/AgX nanostructures can be coupled with other materials to achieve a synergistic antibacterial mechanism.

2.1 Release of silver ions

AgNPs have shown wide-spectrum and effective antibacterial activity against *Staphylococcus aureus* (*S. aureus*; Gram-positive), *Escherichia coli* (*E. coli*; Gram-negative), fungi, and viruses, by releasing silver ions. According to the previous reports, silver ions have been well documented to play bactericidal roles by destroying functional biomolecules such as enzymes, proteins and DNA [26]. Besides, silver ions can destroy cellular metabolic processes, and the released silver ions are able to infiltrate into the bacteria and combine with –SH groups to react with the bacterial proteins, which results in the inactivation of most bacteria [27]. In addition, silver ions can bind to the functional groups of various key enzymes in bacteria, thus hindering bacterial division and other metabolic activities [28]. Silver ions can also bind to DNA and disrupt DNA sequences once they are inside the bacteria cells, and disrupt bacterial metabolism at a genetic level. Furthermore, silver ions can depolarize the cell membrane by binding to protein or phospholipid bilayer of cell membrane, so as to lead to leakage of plenty of bacterial protons, which causes bacterial apoptosis [29]. However, excess amounts of silver ions may cause toxicity to human health, and the aggregation of AgNPs inhibits the antibacterial activity as well.

In order to improve the antimicrobial properties of silver and reduce the safety risk, it is a smart tactic to combine silver with other carrier materials to form silver-doped composites. Subsequently, silver halides were successively used for antibacterial applications, and their solubility in the solution can be tuned by changing the particle size, increasing the light, or adjusting the concentration of the corresponding ions in the solution. However, silver halides have low-solubility [30], so that it is difficult to control the concentration of silver ions, which limit their practical use in antibacterial-associated fields.

The ideal antibacterial agent should not only kill bacteria rapidly, but also prevent bacterial infection over a

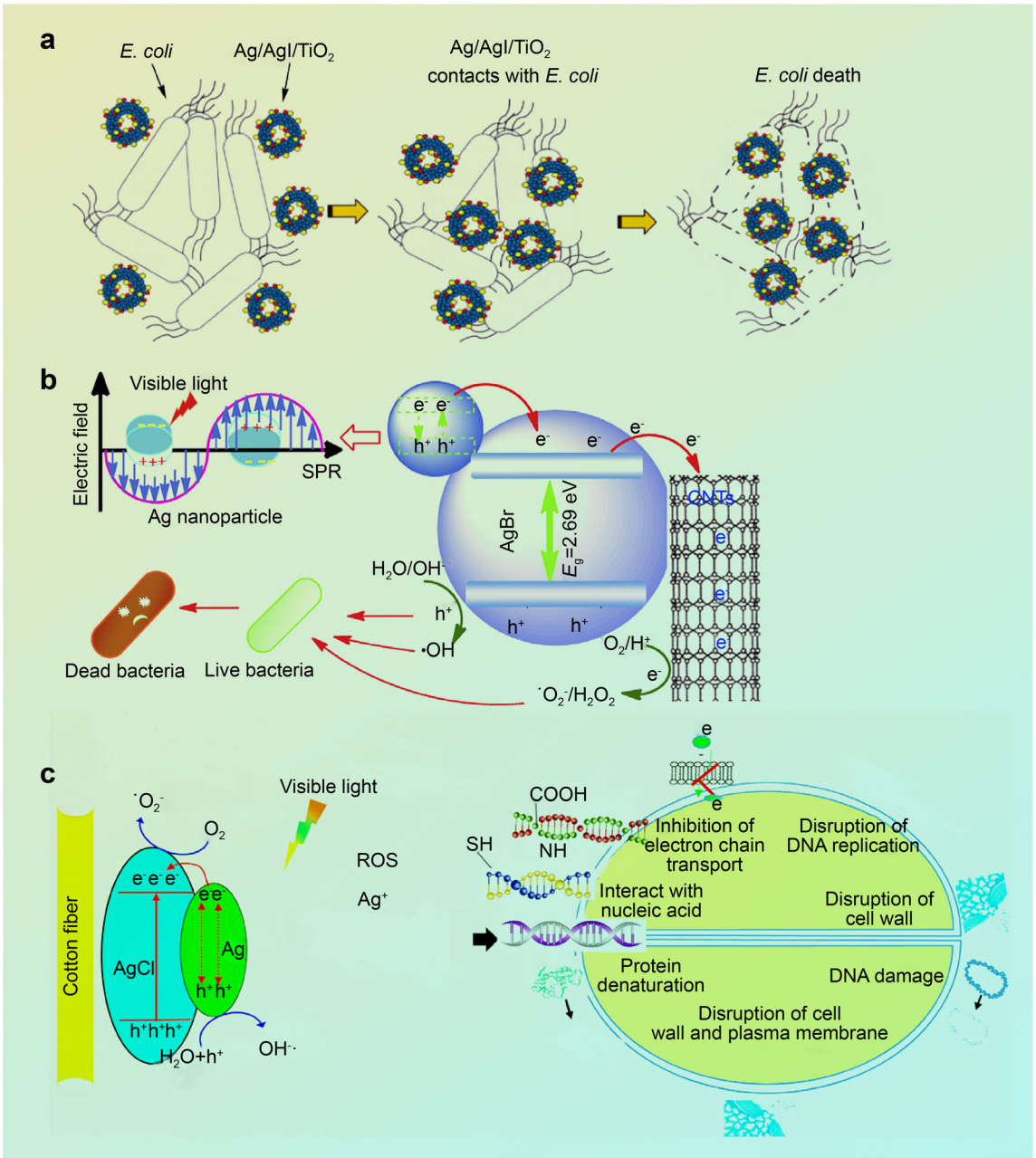

Fig. 1 Illustration of Ag/AgX (X = Cl, Br, I) antibacterial systems. **a** Schematic illustration of inactivation processes of *E. coli* by Ag/AgI/TiO_2. Reproduced with permission from Ref. [40]. Copyright 2020, Springer Nature B.V. **b** photocatalytic inactivation processes and charge transfer of the Ag/AgBr-CNTs photocatalyst under visible light irradiation. Reproduced with permission from Ref. [7]. Copyright 2014, Elsevier. **c** Schematic illustration of solar-photocatalytic disinfection of *E. coli* and *S. aureus* in presence of Ag/AgCl. Reproduced with permission from Ref. [33] Copyright 2019, Elsevier

long time. According to the previous reports, compared with pure nano-silver and pure silver halide nanomaterials, Ag/AgX nanocomposites release silver ions in a controlled and sustained way, thus offering a long-term antibacterial effect. As shown in Fig. 2a, Zhao and coauthors [25] synthesized Ag/AgCl/ABBN composites via impregnation method, and the composites displayed that the open-end hollow tube AgCl might accelerate the separation of electron–hole pairs and enhance the release of ROS, which can effectively control the release of silver ions. The release rate of Ag^+ was prolonged, thus guaranteeing the antibacterial activity. Wu's group synthesized composite nanoparticles (Ag/AgBr/MSNs) with fast and long-term antibacterial activity [31]. The composites were capable of rapid disinfection under simulated VL irradiation and long-term effectiveness by slow release of Ag^+ in the dark for a long time. The amount of released Ag^+ was measured in virto by inductively coupled plasma atomic emission

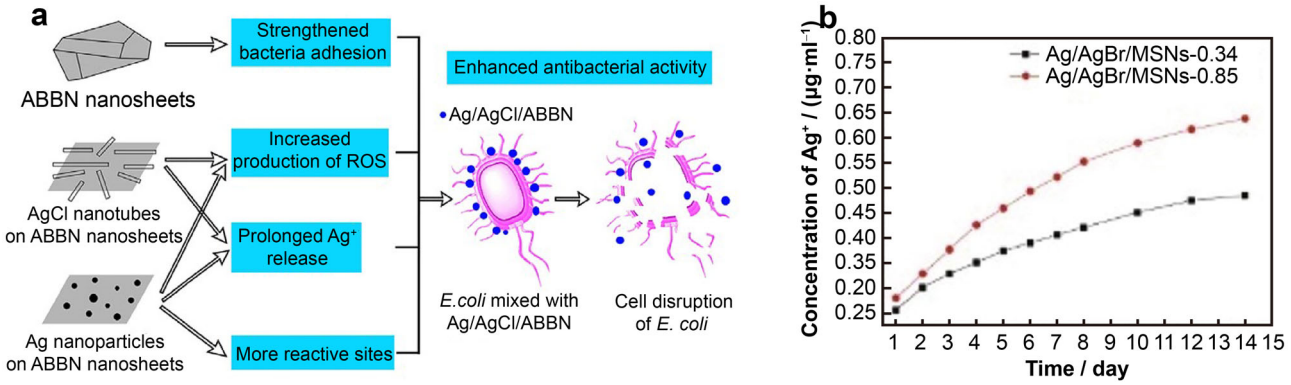

Fig. 2 **a** Schematic illustration of Ag^+ for killing bacteria. Reproduced with permission from Ref. [25]. Copyright 2019, Elsevier. **b** Ag^+ release from Ag/AgBr/MSNs in PBS for 14 days. Reproduced with permission from Ref. [31] Copyright 2018, The Royal Society of Chemistry

spectrometry (ICP-AES). As shown in Fig. 2b, Ag^+ continued to release gradually as a function of aging time during the examined 14 days. The same result was observed in vivo, indicating that Ag/AgBr/MSNs were able to maintain Ag^+ leaching for long-term prevention of bacterial infection. In addition, the long-term effect of bacteria control can stimulate the immune function to produce a large number of white blood cells and neutrophils, which is conducive to promoting wound healing [31]. Then, Wu's group also embedded Ag/Ag@AgCl/ZnO hybrid nanostructures into the hydrogel system to achieve a controlled and long-lasting release of Ag^+ and Zn^{2+} [32]. Under VL irradiation, the Ag/Ag@AgCl/ZnO composite nanostructured hydrogels showed a wide range of antibacterial activities against *E. coli* and *S. aureus* via a synergistic antibacterial model, including ROS and the release of Ag^+ and Zn^{2+}. In the same way, Boufia and co-workers in situ immobilized Ag/AgCl nanoparticles onto cotton fibers [33]. AgNPs reduced the recombination of electron and hole, and improved the interfacial charge transfer process, thus enhancing the photocatalytic efficiency. Free Ag^+ released from AgCl functionalized fabric indicated strong bactericidal effect by inhibiting cell division and destroying the cell membrane and cell contents of bacteria.

To avoid biological toxicity, the interface effect can also be improved by adjusting the morphology of Ag/AgX and combining with other nanomaterials, both of which can effectively control the release behavior of silver ions, and mainly produced ROS or RCS for sterilization [7, 34, 35]. The bactericidal mechanism of reactive species will be described below.

2.2 Generation of reactive species

In comparison with pure silver and silver halides, Ag/AgX nanostructures have stronger photocatalytic activity. Under VL irradiation, the surface of AgNPs based on Ag/AgX composite structure can produce the plasma resonance effect via collective oscillation of electrons which enhances the strong absorption of sunlight and photocatalytic activity. Moreover, AgNPs have good electronic transmission, and make the electron carrier quickly move to the surface of silver halide to inhibit the combination of electrons and holes. Therefore, under light conditions, Ag/AgX produced more active species to show stronger antimicrobial activity due to the enhanced photocatalytic performance.

When a suitable light source is irradiated onto Ag/AgX, the photogenerated electrons (e^-) and holes (h^+) react with O_2 and H_2O, yielding ROS, such as hydroxyl radical ($\cdot OH$), superoxide ($\cdot O_2^-$), singlet oxygen ($^1O_2$), and hydrogen peroxide (H_2O_2) [36]. As shown in Fig. 3, ROS can damage DNA, membrane proteins, lipids and ultimately disintegrate the whole structure of bacteria. For instance, Hou's group [37] found that the antibacterial action of Ag/AgBr/TiO_2 nanotube array electrode against *E. coli* was attributed to generated $\cdot OH$ under VL irradiation (Fig. 4). The reactive species, such as Br^0, $\cdot OH$, $\cdot O_2^-$, and holes, could attack the cell membrane and cell wall of bacteria, disrupt membrane integrity, or destroy the molecules in cell surface. Similar work was reported by Zhou's group, they developed a new Ag-based bactericide through the fabrication of sunlight driven and ultrafine Ag/AgCl anchored on reduced graphene oxide (Ag/AgCl/rGO) [15]. It is worth to mention that the Ag/AgCl photocatalyst is stable enough to release negligible Ag^+, but produces a high amount of ROS to achieve high stability and efficient bacteria-killing efficiency. In contrast to AgX, Ag/AgCl has strong adsorption in the visible region, which is attributed to the plasmon resonance of AgNPs deposited on AgCl particles.

Compared to ROS, the antibacterial activity of RCS, such as chlorine-free radicals and Cl_2, has been rarely reported. In 2020, photo-activatable plasmonic core–shell

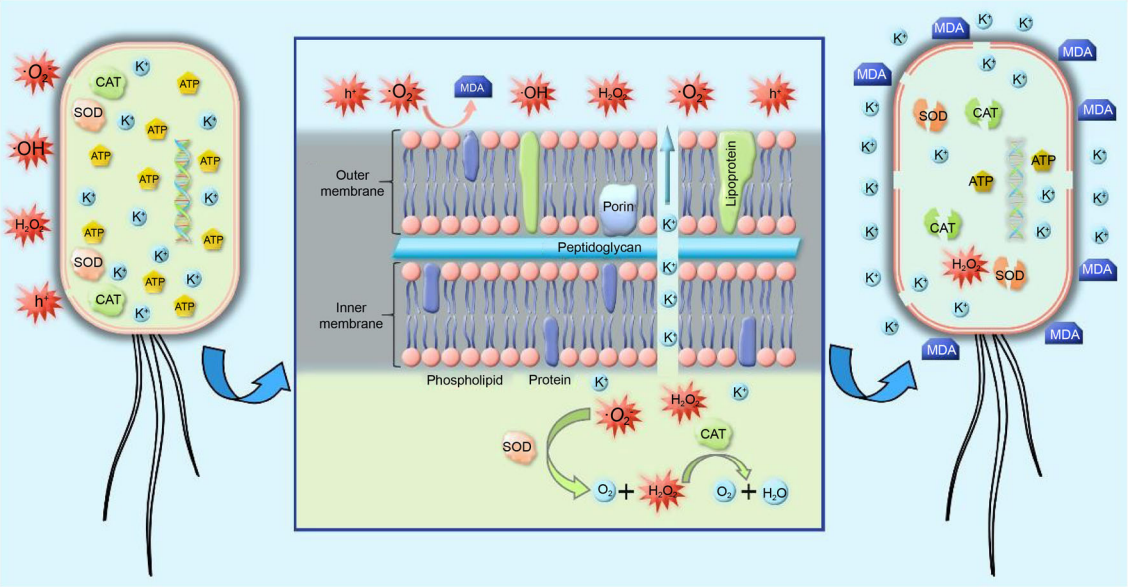

Fig. 3 Illustration summarizing proposed mechanism during photocatalytic disinfection process. Reproduced with permission from Ref. [36]. Copyright 2019, Elsevier

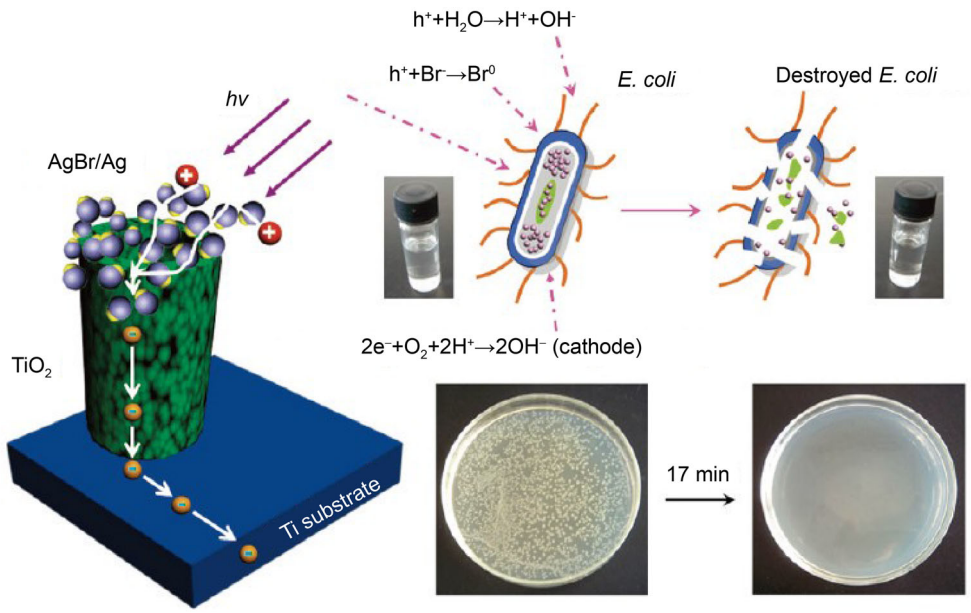

Fig. 4 Inactivation mechanism of Ag/AgBr/TiO_2 nanotube array by hydroxyl radical and antibacterial effect against *E. coli.* Reproduced with permission from Ref. [37]. Copyright 2012, American Chemical Society

Ag@AgCl nanostructures were synthesized through in situ oxidation, and the product has better light absorption ability and efficiently produces RCS through photosensitization reaction ($AgCl + hv \rightarrow Ag^0 + Cl^{\cdot}$) [16]. Under neutral conditions, active chlorine can react with water to produce hydroxyl radical ($Cl^{\cdot} + H_2O \rightarrow Cl^- + {}^{\cdot}OH + H^+$). Then, $Cl^{\cdot}$ and ${}^{\cdot}OH$ fight together against disease-causing drug-resistant bacteria in vivo (Fig. 5), showing an excellent synergistic antibacterial effect against drug-resistant bacteria. In the same year, Bu's group constructed a chlorine-free radical nano-generator with an outer layer of Ag/AgCl heteropoint and an inner layer of nanoparticles (UCNP)/SiO_2 for tumor therapy [38]. As being near-infrared photocatalysis, it no longer depends on ROS, thus Ag/AgCl can produce chlorine-free radicals to highly improve the therapeutic effect in an anoxic environment.

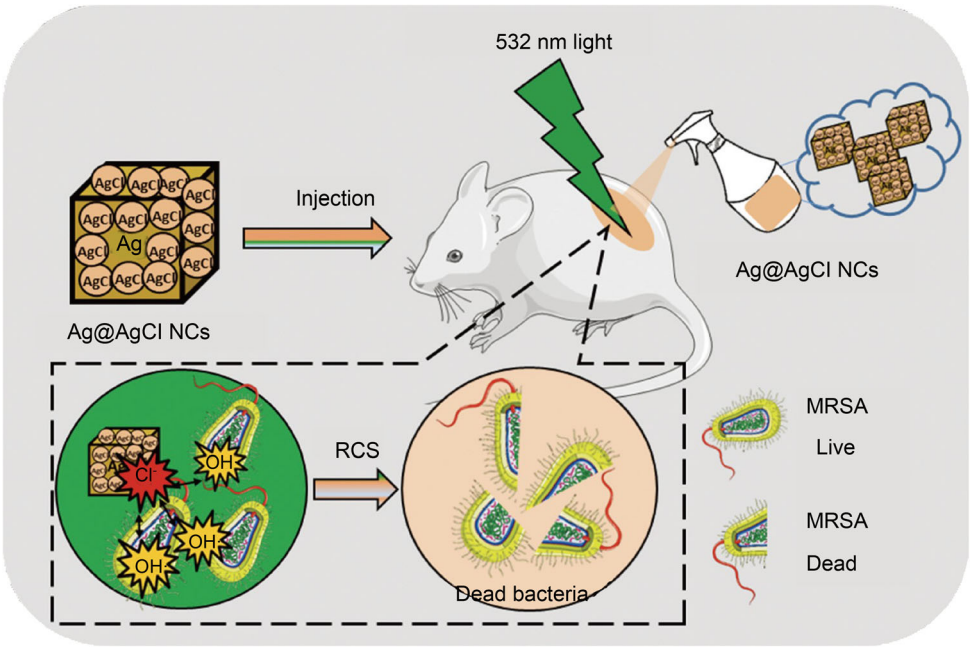

Fig. 5 Schematic representation of in vivo experimental design of chlorine and hydroxyl radicals mediated phototherapeutic effect for killing drug-resistant bacterial models on infected skin. Reproduced with permission from Ref. [16]. Copyright 2020, The Royal Society of Chemistry

2.3 Combined mechanism

The synergistic antibacterial mechanism of Ag/AgX nanostructures can be divided into two categories. Firstly, the released Ag^+ combines with ROS generation under light conditions. ROS produced by photocatalyst could successfully destroy bacteria under VL irradiation, and Ag^+ can change the permeability of cell membrane and denaturation proteins of bacteria. Secondly, Ag/AgX nanostructures can be coupled with other materials, such as carbon materials, metal oxides, and semiconductors, to show synergistic antibacterial effects [25, 32–37, 39–44].

As an important member of the carbon material family, reduced graphene oxide (RGO) can not only be used as an electron transfer medium to greatly reduce the coincidence rate of photogenerated holes, but also form a lipopolysaccharide bridge bond, which is easy to combine with the surface of microbial cells to improve the photocatalytic efficiency through adsorption. Xia and co-workers [35] prepared a series of Ag/AgX-RGOs (X = Cl, Br, I) composites by coupling graphene sheets with plasma photocatalysis and explored their bactericidal mechanisms (Fig. 6). Their study proved that the antibacterial activity of Ag/AgX-RGO_S was enhanced via the synergistic activities of three components, and the proposed mechanisms contained chemical attraction of RGO, followed by bactericidal effect of released Ag^+ species and primary oxidative stress of plasmon-induced H_2O_2, leading to the damage of microbial metabolism processes, the destruction of cell envelope, and the leakage of intracellular substances. It was further proved that the bactericidal order is Ag/AgBr-RGO > Ag/AgCl-RGO > Ag/AgI-RGO, because the narrow band gap of AgBr is easier to be activated by a wide range of VL.

Wu et al. designed and synthesized Ag/AgBr-loaded mesoporous silica nanoparticles (Ag/AgBr/MSNs) for rapid sterilization and accelerated wound healing under VL irradiation [31]. Since Ag/AgBr nanostructures are prone to generating electron–hole pairs after absorbing light, ROS production increases the antibacterial activity (Fig. 7). Under simulated sunlight irradiation, the killing rates of Ag/AgBr/MSNs against *S. aureus* and *E. coli* were 95.62% and 99.99% after treatment for 15 min, respectively. They also confirmed that the composite inhibits bacterial growth and destroys bacterial membranes through electrostatic interaction. The release of Ag^+ can further effectively prevent bacterial infection for a long time and stimulate the immune function to generate a large number of white blood cells and neutrophils, which are able to accelerate the wound healing process.

Zinc oxide (ZnO) nanoparticles have attracted increasing interest and have been widely employed as a commercial antibacterial material due to the low-cost, chemical stability, and good biocompatibility [45]. Compared with traditional photocatalytic antibacterial agents, ZnO has a narrower band gap and better photocatalytic properties, but it still suffers from rapid recombination of electrons and holes. Thus, combination of ZnO with other photocatalysts is undoubtedly an alternative strategy. Yu et al. [41]

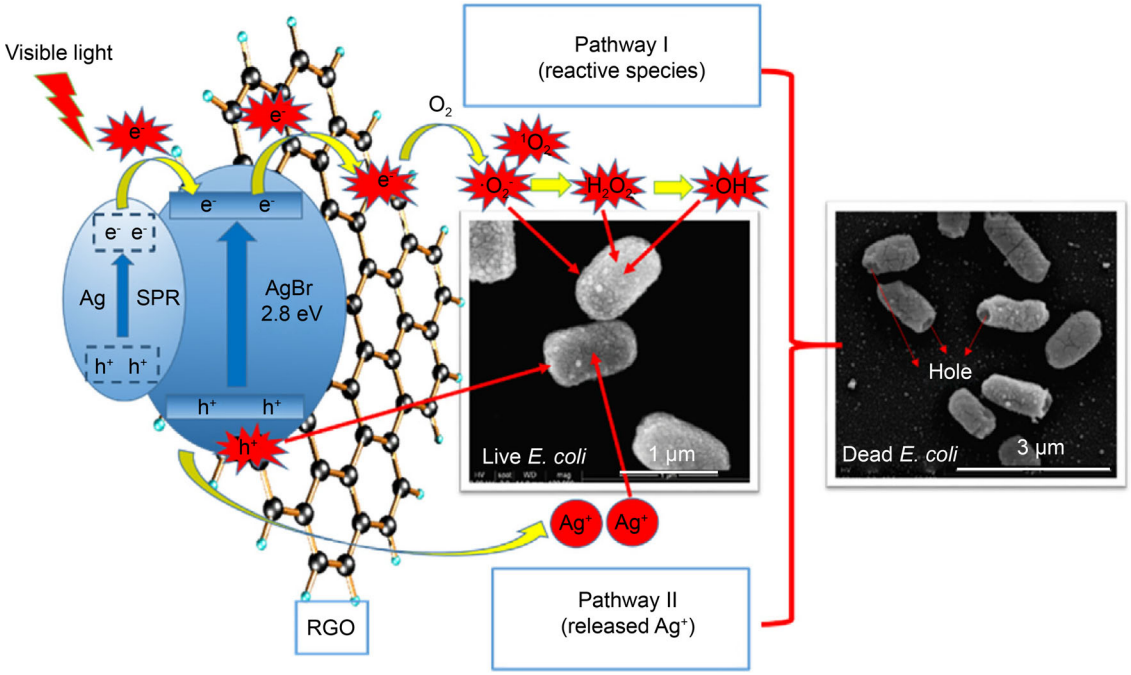

Fig. 6 Proposed synergistic photocatalytic bacterial inactivation mechanism by plasmonic Ag-AgBr/0.5% RGO composite photocatalyst. Reproduced with permission from Ref. [35]. Copyright 2016, Elsevier

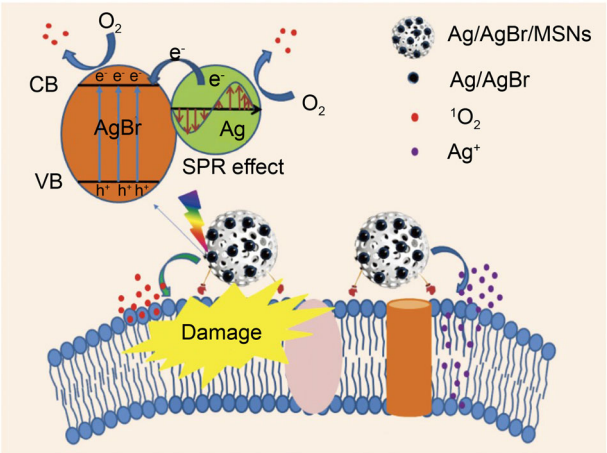

Fig. 7 Schematic illustration of bacteria-killing through photodynamic effects of Ag/AgBr/MSNs under VL and innate antimicrobial ability of Ag^+. Reproduced with permission from Ref. [34]. Copyright 2018, The Royal Society of Chemistry

prepared Ag@AgCl/ZnO nanocomposites using pectin (CEP) as matrix material through a two-step method. Under VL irradiation, ROS, released Zn^{2+} and Ag^+ worked together to achieve a synergistic antibacterial activity. The photocatalytic antibacterial activity of the CEP-Ag@AgCl/ZnO nanocomposite is illustrated in Fig. 8. The nanocomposite indicated a promising future application in food packaging, sterilization, and medical disinfection.

Among all kinds of semiconductor materials, TiO_2 has been widely studied due to its non-toxicity, high chemical stability and efficiency in degrading organic pollutants, and bacteria-killing ability in water treatment. However, TiO_2 has two disadvantages: one is the poor solar efficiency due to its wide band gap; the other is the low quantum yield that arises from the rapid recombination of photo-generated electrons and holes [46]. To improve its potential for practical use, the combination of TiO_2 with AgCl@Ag is an alternative tactic. The sandwich-structured AgCl@Ag@TiO_2 plasmonic photocatalyst succeeded in wastewater treatment, sterilization, and other photocatalytic fields. The possible mechanism is based on seven equations as following, it is obvious that $\cdot O_2^-$, $\cdot OH$, and Cl^0 are the main factors for pollutant degradation and bacterial decontamination.

$$\text{Ag-NPs} + h\nu \rightarrow \text{Ag-NPs}* \rightarrow \text{Ag-NPs} + \text{Ag-NPs}^{\oplus} \quad (1)$$

$$\text{Ag-NPs}-\text{TiO}_2 + \text{O}_2 \rightarrow \text{Ag-NPs}-\text{TiO}_2 + \cdot\text{O}_2^- \quad (2)$$

$$\text{Ag-NPs}^{\oplus} + \text{Cl}^- \rightarrow \text{Ag-NPs} + \text{Cl}^0 \quad (3)$$

$$\text{Ag-NPs}^{\oplus} + \text{OH}^- \rightarrow \text{Ag-NPs} + \cdot\text{OH} \quad (4)$$

$$\text{Cl}^0 + \text{Organics/bacteria} \rightarrow \text{Degradation products} + \text{Cl}^- \quad (5)$$

$$\cdot\text{O}_2^- + \text{Organics/bacteria} \rightarrow \text{Degradation products} \quad (6)$$

$$\cdot\text{OH} + \text{Organics/bacteria} \rightarrow \text{Degradation products} \quad (7)$$

In recent years, synergistic antimicrobial systems in the basis of combining photodynamic therapy (PDT) and

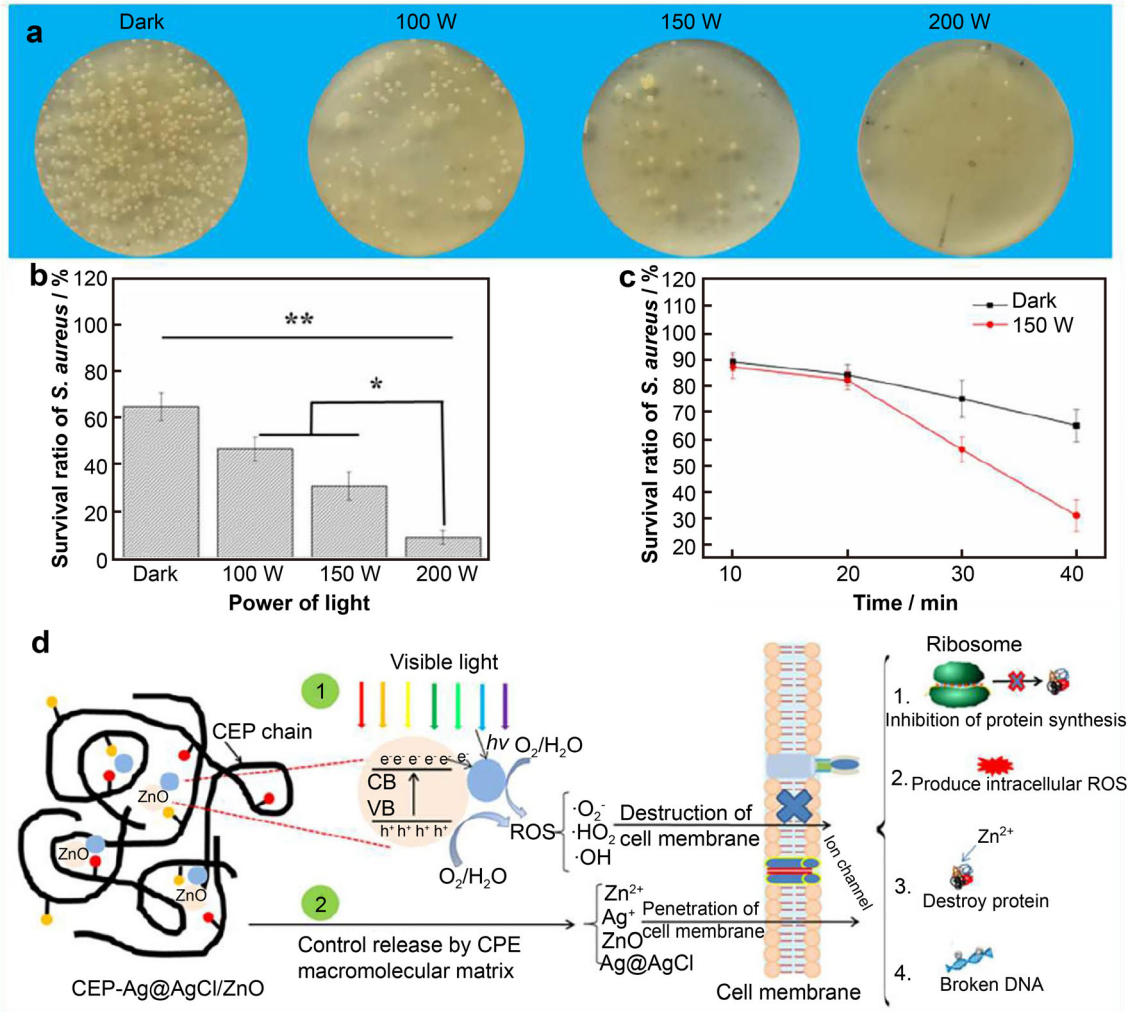

Fig. 8 Schematic illustration of preparation and application of CEP-Ag@AgCl/ZnO nanocomposites as visible light triggered antibacterial agents. **a** Photographs of *S. aureus* colonies, and **b** survival ratios of *S. aureus* after treatment with CEP-Ag@AgCl/ZnO nanocomposites and simulated sunlight with different powers for 40 min; **c** simulated sunlight at 150 W for different times; **d** schematic illustration of possible antibacterial mechanism of CEP-Ag@AgCl/ZnO nanocomposites. Reproduced with permission from Ref. [41] Copyright 2019, Elsevier

photothermal therapy (PTT) have been widely explored [47]. PDT produces ROS to kill bacteria rapidly by damaging the cell membrane, and PTT involves the conversion of solar energy into heat energy under light irradiation. In 2019, Wu's research group embedded Ag/AgCl nanostructures in hydrogels system containing polyacrylamide (PAM) and polydopamine (PDA) [39]. Under VL irradiation, Ag/AgCl excited electrons, O_2, and H_2O in the environment to capture electrons and form bactericidal ROS. PAM-PDA had good photothermal preservation, which improved local temperature and the permeability of bacterial membrane, so that ROS was more sensitive to the inner membrane, and smoothly entered the bacterial inside. The synergistic effect of PTT and PDT was realized by destroying the bacterial structure and metabolic function, as shown in Fig. 9.

3 Nanoengineering of Ag/AgX binary system

Past few decades witnessed the great advances in nanotechnology due to specific properties of the nanoparticles, such as large surface areas and high responsiveness [48–50]. Some functional nanoparticles showed significant potential for antibacterial applications [51–54]. AgNPs have been used as the most popular antibacterial agents in many fields, such as food packaging and medical devices [31]. Unfortunately, some disadvantages of AgNPs occurred during the applications. First, it is a challenge to maintain a stable flux of high concentration of silver ions, which is the key to the antibacterial function. Besides, due to the aggregation and oxidation of AgNPs in the solution, their instability significantly affects the antibacterial ability. Third, AgNPs show some toxicity to human health and

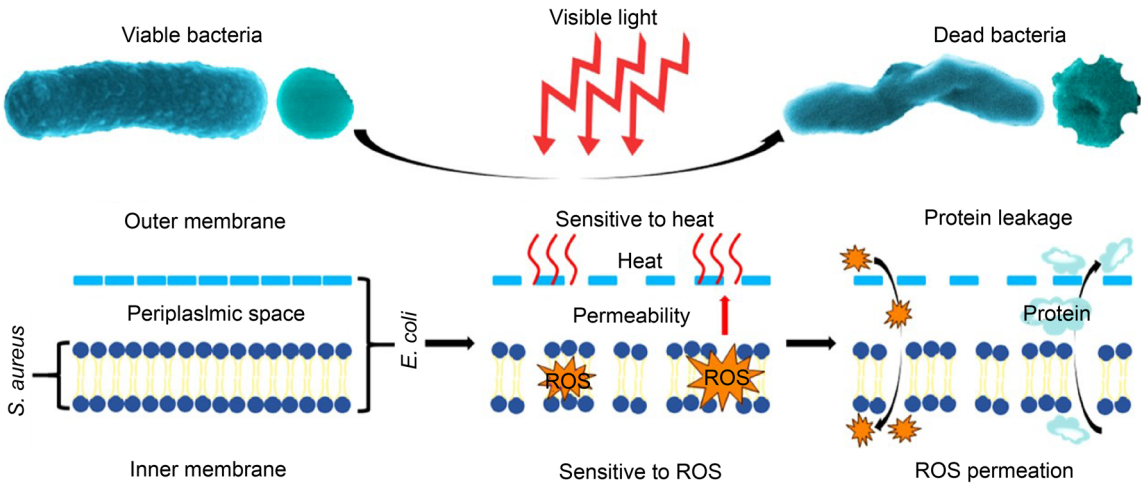

Fig. 9 Antibacterial mechanism and process of disrupting bacterial membrane. Reproduced with permission from Ref. [39]. Copyright 2019, American Chemical Society

living environment, limiting their practical applications. In contrast to AgNPs, AgX maintains the continuous release of high flux silver ions. However, AgX is easily transformed into Ag^0 and loses the photocatalytic activity due to its poor photostability under light irradiation, which hinders its practical applications [55]. Therefore, much more efforts have been devoted to designing Ag/AgX (X = Cl, Br or I) nanostructures and related composite materials, which enhances the absorption of VL and provides new opportunities to develop VLD photocatalysts. Furthermore, recent studies proved that Ag/AgX exhibited very high antibacterial ability under light irradiation because of their narrow band gaps, available Schottky barrier, and excellent stability [25]. Ag/AgX nanostructures have also been explored as a photostable photocatalyst with high VL photoactivity [56]. In addition, Ag/AgX nanostructures have been coupled with other nanomaterials to improve photoactivity and achieve highly efficient antibacterial agents.

The Ag/AgX system shows high antibacterial ability under light irradiation; however, it still suffers from high recombination rate of photo-generated electron–hole pairs, low photocatalytic efficiency, and poor stability [6, 7, 57–60]. When the illumination time is longer, the AgX surface will deposit excess silver, which both reduces the photocatalytic activity and bactericidal efficiency. In order to solve these drawbacks, researchers mainly focused on the nanoengineering of Ag/AgX binary system [24, 61]. To date, the nanoengineering of Ag/AgX binary system can be classified generally into the following three categories [62]: (i) carbon materials, such as graphite [63], graphene [64–66], g-C_3N_4 [67]; (ii) metal oxides, such as Fe_2O_3 [56], TiO_2 [67–69], Cu_2O [70, 71], ZnO [72], and WO_3 [55, 73]; (iii) double oxides, such as $CaTiO_3$ [74], $LaFeO_3$ [75], $ZnFe_2O_4$ [76], and MoS_2 [77]. These nanostructures can provide more reactive sites for Ag/AgX reacting with thiol groups (-SH) of bacteria and result in inactivating the bacterial proteins by damaging the cell membrane integrity. It has been reported that Ag/AgX@GO nanocomposite has significantly enhanced photocatalytic activity and stability, compared to those of bare Ag/AgX (X = Cl, Br) plasmonic photocatalysts [64–66]. In another report, Ag/AgCl/$ZnFe_2O_4$ composites demonstrated an enhanced photocatalytic performance, which was attributed to the extended light-response range and increased separation efficiency of electron–hole pairs through the introduction of $ZnFe_2O_4$ into Ag/AgX system. As a result, Ag/AgCl/$ZnFe_2O_4$ composites exhibited superior antibacterial activity than pristine Ag/AgCl under VL irradiation [76]. In order to improve antibacterial and photocatalytic activities, Jing et al. [40] designed and synthesized Ag/AgI/TiO_2 heterostructures with unique morphology, hollow structure, and high surface area. The results showed that the heterostructures had higher antibacterial activity against *E. coli* and high photocatalytic activity toward RhB under UV and VL irradiation. Cui et al. [78] synthesized a 1-D core–shell structure Fe-Ag@AgCl by pulsed electrodeposition strategy via using polycarbonate (PC) template followed by in situ oxidation with $FeCl_3$ (Fig. 10). The final product exhibited excellent photocatalytic performance by inactivating 1×10^7 CFU·ml^{-1} of *E. coli* after 120-min VL irradiation.

4 Factors impacting antibacterial efficiency of Ag/AgX nanostructures

Numerous strategies have been developed for synthesizing Ag/AgX (X = Cl, Br, I) based photocatalysts to increase the surface area and band alignment by coupling with other

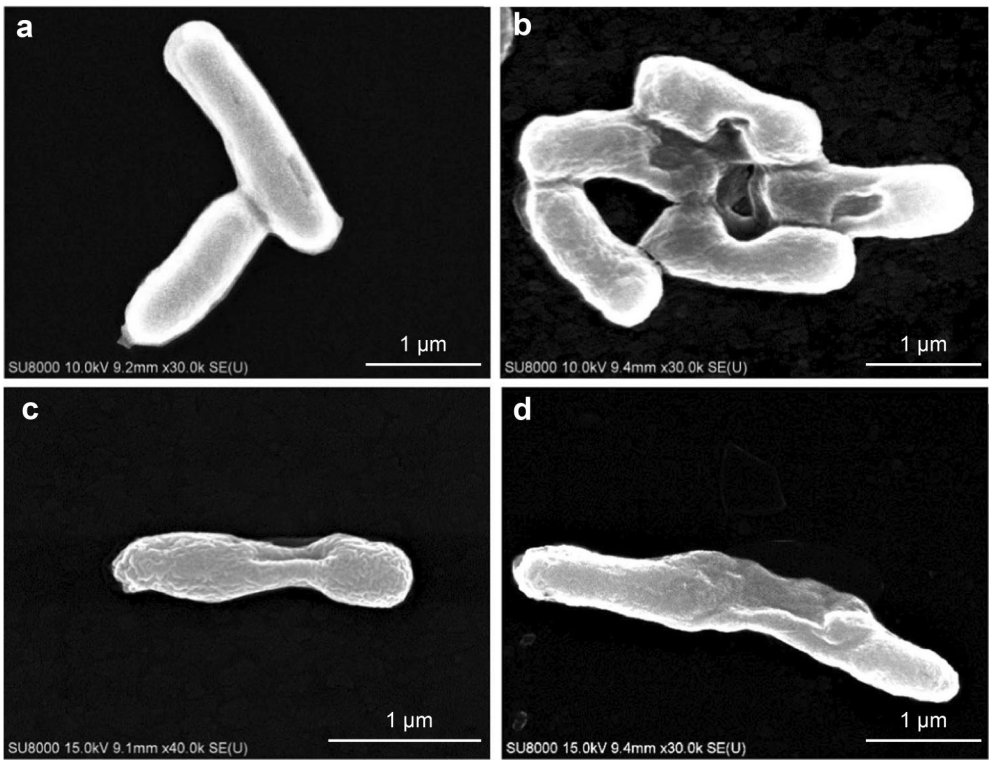

Fig. 10 SEM images of *E. coli* during photocatalysis treatment with Fe-Ag@AgCl under VL irradiation at **a** 0 min, **b** 40 min, **c** 80 min, and **d** 120 min. Reproduced with permission from Ref. [78]. Copyright 2020, Springer

semiconductors [79]. Although Ag/AgX shows a good potential application in the antibacterial field, there are some difficulties in its practical use due to its severe aggregation and photo corrosion. Herein, we discussed some factors that influence the antibacterial efficiencies of Ag/AgX nanostructures. The antibacterial mechanism of Ag/AgX nanostructures is briefly shown in Fig. 11.

4.1 Size impact

It is known that the size of catalysts affects their adsorptive ability: a smaller size promotes the catalytic performance. At the same time, compared to those with their bulk counterparts, the catalysts with nanosize indicate high surface-to-volume ratio and have more active sites, which accelerate the catalytic reaction [41]. For example, in Urakaev's report, AgNPs about 7 nm in diameter were bound onto AgCl via the photoreduction of AgCl (Ag^{+}) to Ag^{0} atoms (Fig. 12). AgCl/Ag CNPs strongly and uniformly absorb light in the visible region, which provides themselves with high catalytic activity in the photodegradation of methylene blue dye under the influence of light of neon tubes [62]. Zhu et al. [80] synthesized a series of Ag/AgCl/GO nanostructures with different sizes by introducing GO nanosheets into the Ag/AgCl system. They came to the following conclusion: (i) compared to the bare Ag/AgCl nanospecies, the Ag/AgCl/GO displayed enhanced catalytic activities; (ii) for similar shape of nanospecies, those with a smaller size showed higher catalytic performances; and (iii) for similar size of nanoarchitectures, the cube-like nanospecies exhibited higher catalytic activities as their near-spherically shaped counterparts. These results suggest that the photocatalytic performances of Ag/AgCl are dependent on the size of nanostructures. Zhou et al. [15] developed an Ag/AgCl/rGO bactericide by increasing the content of Ag^{0} and reducing the particle size. The results showed that the size of Ag/AgCl NPs in the nanomaterial is ultrafine ($\sim$4 nm); meanwhile, the surface mole ratio of the metallic Ag^{0} to Ag^{+} in the nanomaterial is very high (2.11:1). The Ag/AgCl/rGO nanomaterial not only possessed an excellent bactericidal property against both Gram-positive and Gram-negative bacteria, but also exhibited superior stability [81].

4.2 Morphology impact

It has been reported that the morphology of photocatalyst is an important factor to influence the photocatalytic performance [82]. Recently, Ag/AgX photocatalysts with various structures, such as porous nanocomposite [83], cube-like,

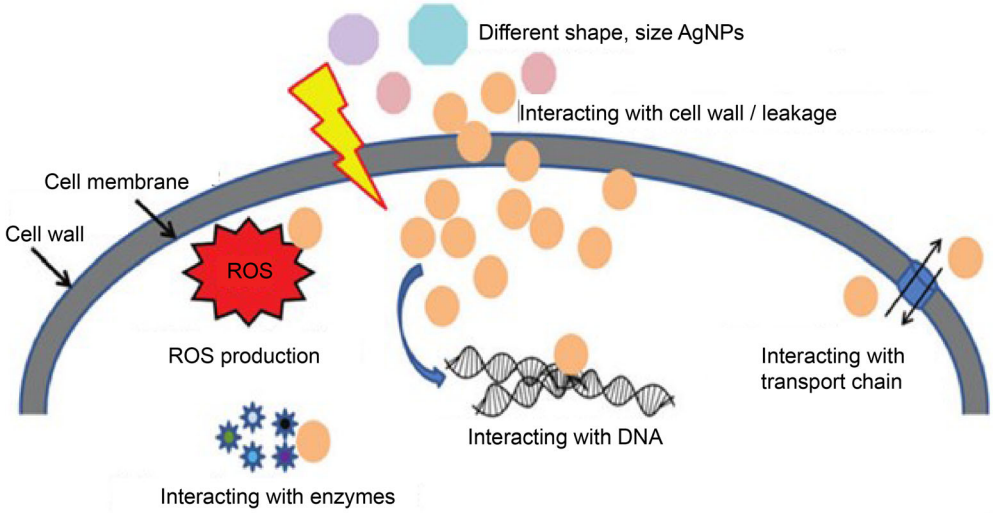

Fig. 11 Possible bactericidal mechanisms of Ag/AgX. Reproduced with permission from Ref. [42]. Copyright 2017, The Royal Society of Chemistry

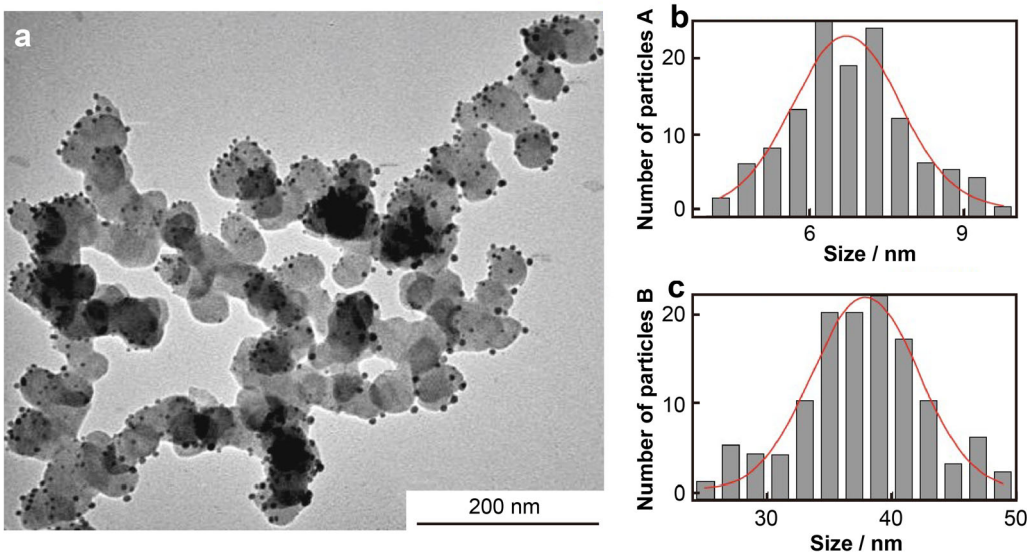

Fig. 12 **a** TEM image of a AgCl/Ag CNP sample; corresponding size distributions of **b** Ag and **c** AgCl nanoparticles. Reproduced with permission from Ref. [62]. Copyright 2020, Springer Nature B.V

necklace-like [84], and near-spherical nanocatalysts [85], have been synthesized and their VL photocatalysis has been investigated [86].

Most synthetic methods of Ag/AgX follow the reduction route. That is, the structure of AgX crystal is prepared first, and then part of the AgX sample is converted to Ag by photoreduction or polyol reduction. However, this method can only obtain a single morphology, and the ratio of Ag/AgX is difficult to control. Nowadays, core–shell structured nanomaterials have attracted particular research attention because of their great potentials in the protection, modification, and functionalization of core particles, with suitable shell materials to enable specific physical, chemical, and biological performance. For instance, Bi and Ye [87] demonstrated a general in situ oxidation strategy to synthesize uniform Ag/AgCl core–shell nanowire heterostructures from Ag nanowires, using $FeCl_3$ as an oxidant at room temperature, as shown in Fig. 13a. The photocatalytic performance examination for the decomposition of methylene orange (MO) indicated that the unique core–shell nanowires exhibited high catalytic activities under VL illumination. Since then, in situ oxidation has been widely used to prepare Ag/AgCl core–shell nanostructures. The simplest strategy is using Ag nanostructures with a specific morphology as a template, such as nanowires, nanorods, cubes, spheres, triangular plates. For

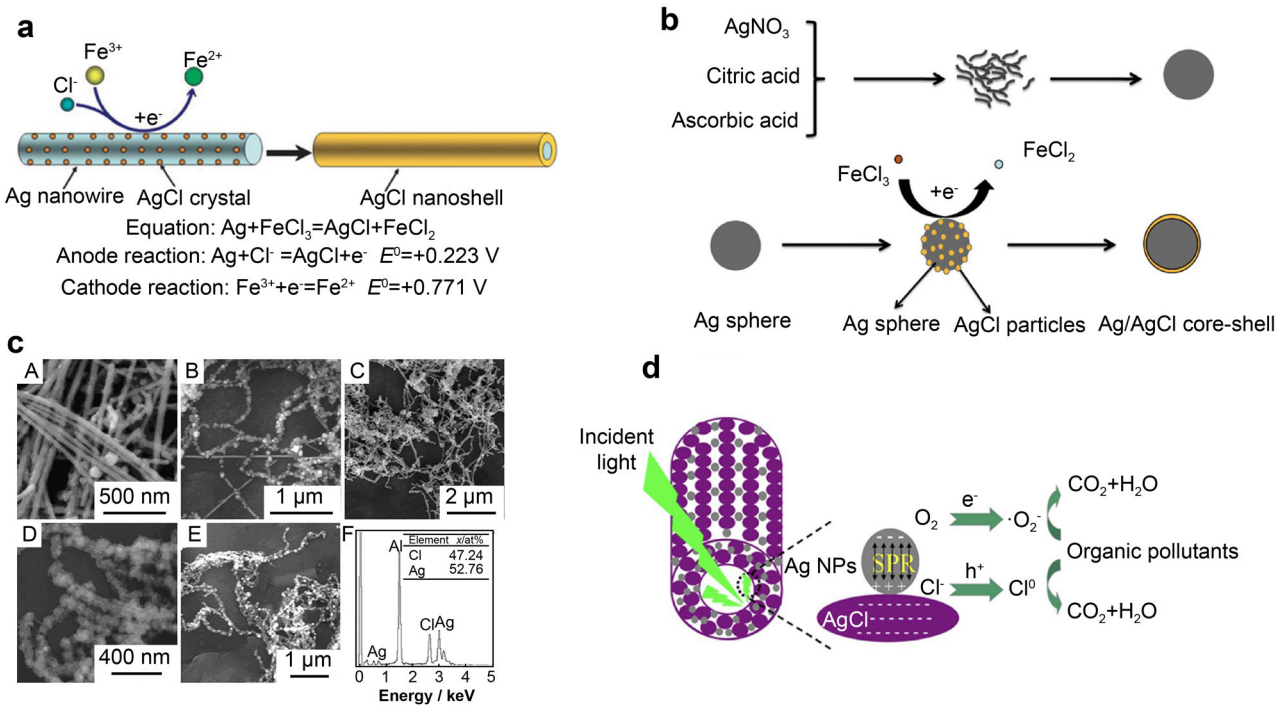

Fig. 13 Core–shell structures with different morphologies. **a** Ag/AgCl core–shell nanowires. Reproduced with permission from Ref. [87]. Copyright 2009, The Royal Society of Chemistry. **b** Core–shell Ag/AgCl sphere material. Reproduced with permission from Ref. [85]. Copyright, 2012 Elsevier B.V. **c** Necklace-like Ag/AgCl. Reproduced with permission from Ref. [84]. Copyright 2014, Wiley–VCH. **d** Ag@AgCl nanotubes. Reproduced with permission from Ref. [86]. Copyright 2014, American Chemical Society

example, using in situ oxidation reaction, Ma et al. [85] synthesized a plasmonic photocatalyst Ag/AgCl core–shell sphere by a facile and effective method via an oxidation reaction between Ag spheres and $FeCl_3$, as shown in Fig. 13b. The obtained Ag/AgCl is an active and stable VLD plasmonic catalyst, which serves as a promising candidate for practical use for the degradation of hazardous pollutants in water. Huang's group developed Ag/AgCl necklace-like nano-heterostructures with an average diameter of 55 nm by a facile in situ oxidation process, as shown in Fig. 13c [84]. The photocatalytic investigations demonstrated that the Ag/AgCl heterostructures with an 85% AgCl component have an excellent activity for the decomposition of organic pollutants and water splitting to produce oxygen. As shown in Fig. 13d, Sun et al. [86] reported a facile process for synthesizing stable and hollow plasmonic photocatalyst Ag@AgCl nanotubes via a template-based method. The as-prepared Ag@AgCl nanotubes exhibited excellent photocatalytic performance and high stability. Based on the proposed mechanism, the improved photocatalytic activities of the Ag@AgCl hybrids can be ascribed to the enhanced surface area for dye molecule

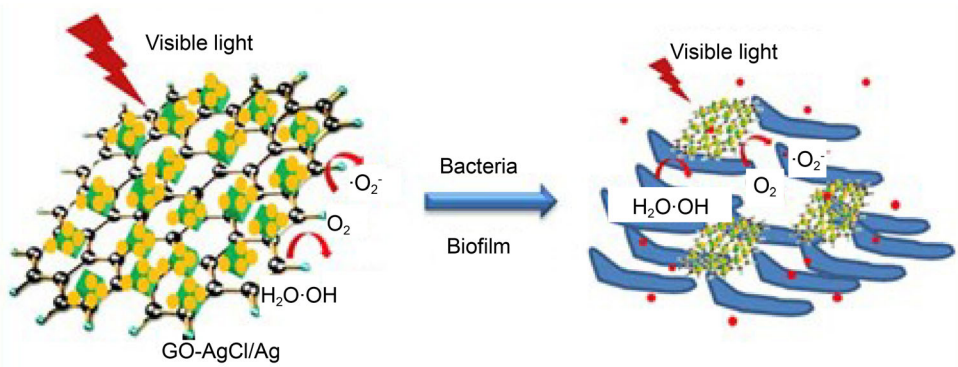

Fig. 14 Schematic illustration of enhanced antibacterial and biofilm-disrupting effects of GO-AgCl/Ag nanocomposites under VL irradiation. Reproduced with permission from Ref. [64]. Copyright 2017, Elsevier

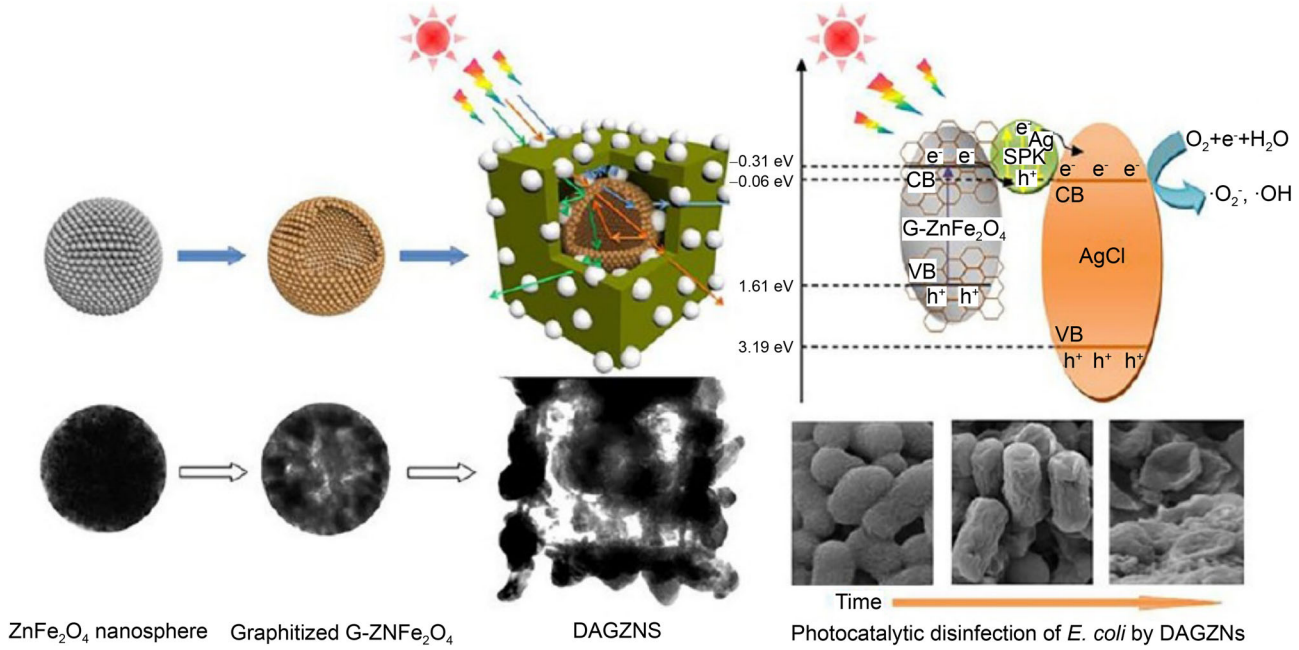

Fig. 15 Schematic illustration of double-shell Ag/AgCl/G-$ZnFe_2O_4$ nanocube with enhanced light absorption and superior photocatalytic antibacterial activity. Reproduced with permission from Ref. [94]. Copyright 2020, American Chemical Society

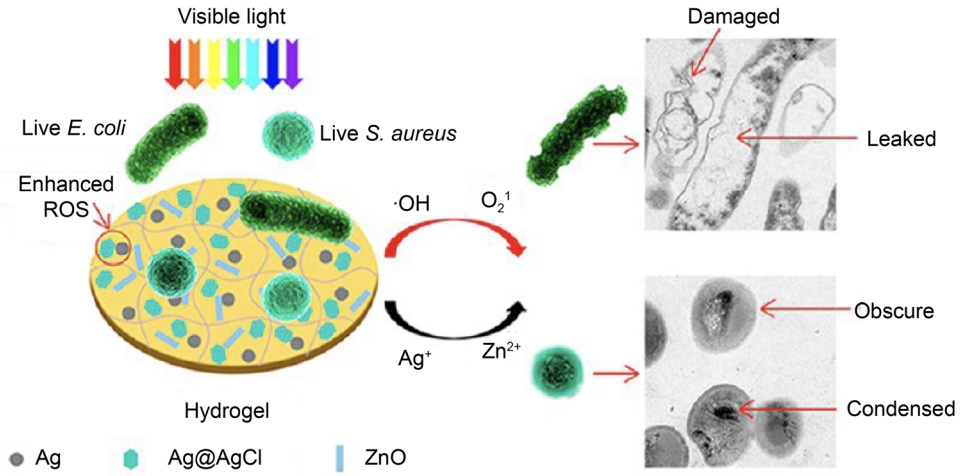

Fig. 16 Schematic illustration of a hybrid hydrogel embedded with Ag/Ag@AgCl/ZnO hybrid nanostructures showing enhanced antibacterial activity. Reproduced with permission from Ref. [32]. Copyright 2017, American Chemical Society

adsorption, VL absorbance, and the efficient charge separation of the hybrid nanostructures.

4.3 Impact of surface chemistry

Compared with solid and spherical particles, photocatalysts with hollow or mesoporous structures have attracted increasing attention because of their high specific surface area and enhanced light absorption capacity. As is well-known, the charge separation/transfer is another crucial issue affecting the catalytic performance of a photocatalyst. The Ag/AgX-based composite photocatalysts have been confirmed that themselves are not only photostable photocatalysts with high VL photoactivity but also can be used to couple with other semiconductors to improve the photoactivity [56]. So far, many Ag/AgX-based composite photocatalysts have been fabricated [7, 46, 88–90], such as Ag/AgCl/$W_{18}O_{49}$, Ag/AgCl/ZnO, Ag/AgX/GO (X = Cl, Br), Ag/AgBr/TiO_2, Ag/AgCl/BiOCl [91], and AgCl@Ag@TiO_2. These composites displayed high

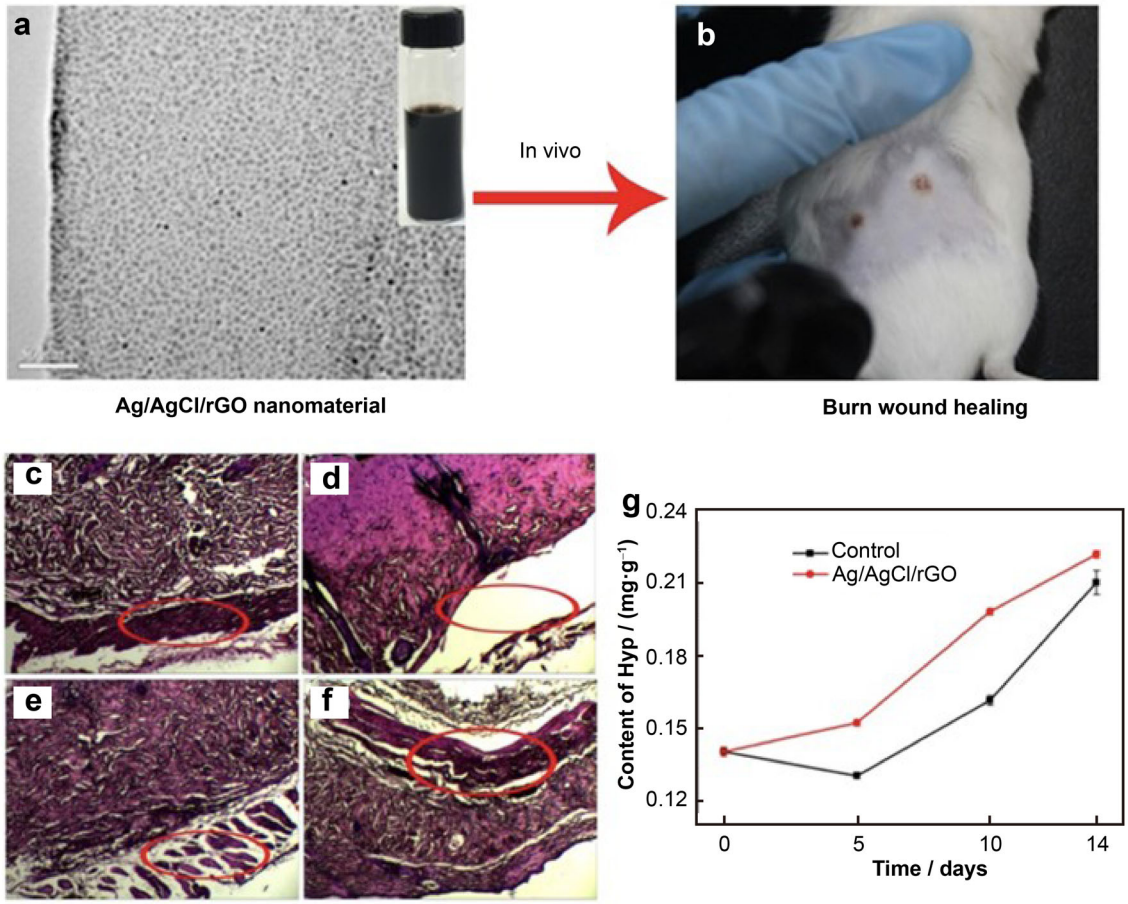

Fig. 17 **a, b** Schematic illustration of Ag/AgCl/rGO nanomaterial in burn wound healing; **c** healthy mouse skin, **d** initial burned skin of mice, and burned skin of mice treated with **e** water and **f** Ag/AgCl/rGO on day 10; **g** contents of hydroxyproline in granulation tissues of skin wounds in different healing periods. Reproduced with permission from Ref. [15]. Copyright 2016, American Chemical Society

photocatalytic activity in the photodegradation of organic pollutants and high antibacterial ability under light irradiation. It has been proved that, with the increasing generated ROS and long-term released Ag^+, antibacterial activities can be ensured [92, 93]. Besides, some two-dimensional nanosheets have been chosen as the support materials to improve ROS production and prolong the release of Ag^+. For instance, Wang et al. [64] modified GO nanosheets with AgCl/Ag nanocomposite, and the product showed high VL absorption and high antibacterial property (Fig. 14).

Zhang et al. [94] employed a self-etching method to fabricate a hollow G-$ZnFe_2O_4$ nanosphere to carry and functionalize with polydopamine (PDA), which is used as the template and reductant to prepare double-shell Ag/AgCl/G-$ZnFe_2O_4$ nanocubes (DAGZNs) photocatalytic (Fig. 15). The antimicrobial activity of DAGZNs under VL irradiation showed high bactericidal properties against Gram-positive bacterium *S. aureus* and Gram-negative bacterium *E. coli,* as well as excellent recyclability.

5 Antibacterial applications of Ag/AgX nanostructures

Since fighting against bacterial infections becomes great challenging to modern healthcare system, there is an urgent requirement to develop more effective and powerful antibacterial agents to address the growing microbial threats. In the past few decades, Ag/AgCl functioned as an antibacterial agent, and had made great progress in practical applications, especially in wound healing and water disinfection [95, 96].

5.1 Wound healing

Wound infection is a common danger to human health [97] and in particular often caused by bacterial infections. Once the wound was infected, the recovery would be slowed and usually accompany some other complications, resulting in more pain for the patient. Antibiotics are the most effective for disinfection on the infected wound, but they cannot kill

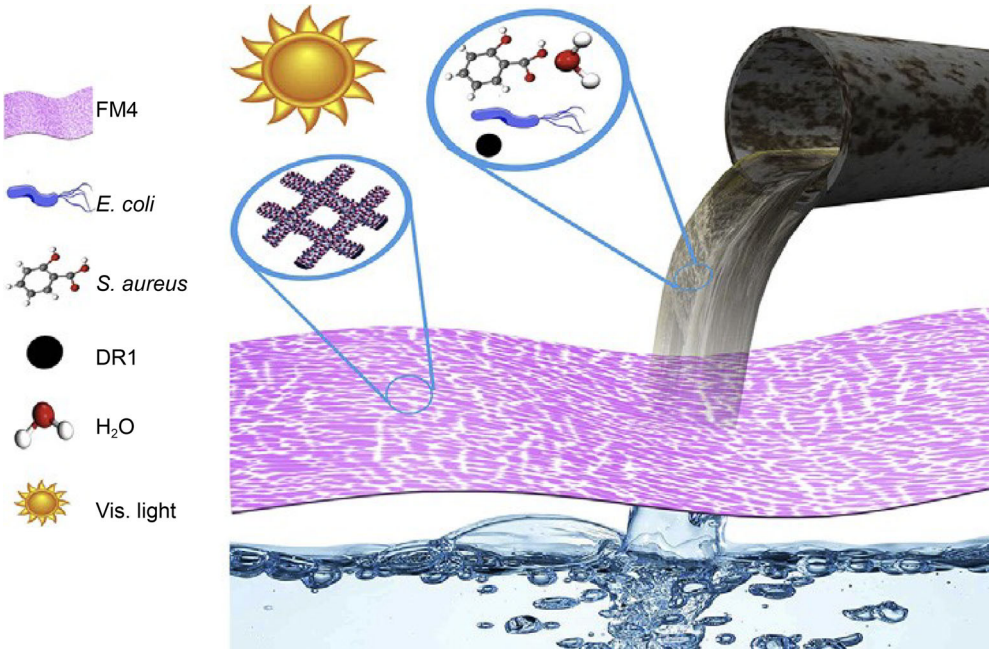

Fig. 18 Schematic diagram of efficient wastewater purification by hydrophilic electrospinning PAN/AgBr/Ag fiber membrane. Reproduced with permission from Ref. [107]. Copyright 2018, Elsevier B.V

bacteria instantaneously. Furthermore, the overuse of antibiotics may to some extent lead to bacterial resistance, even the occurrence of "superbugs". Thus, an alternative antibacterial strategy is highly required [34]. Wu's group constructed a hydrogel composite incorporated with carboxymethyl cellulose (CMC) and Ag/Ag@AgCl/ZnO hybrid nanostructures that showed excellent photocatalytic activity and broad antibacterial efficiency against both of Gram-positive and Gram-negative bacteria under VL irradiation (Fig. 16) [32]. In addition, hydrogels can be triggered by changing the environment's pH and promoting the immune system to accelerate wound healing.

Ag/AgCl/rGO nanomaterials also have been used for wound healing. Typically, Zhou et al. [15] demonstrated a long-term and highly efficient antibacterial nanomaterial made of reduced graphene oxide (rGO) sheets coated with high-quality ultrafine Ag/AgCl (Fig. 17). After the treatment with Ag/AgCl/rGO nanomaterial, the burn wound showed a fast healing rate and epidermis regeneration. This suggested that Ag/AgCl/rGO nanomaterial displayed an efficient antibacterial performance and was beneficial on burn wound healing.

5.2 Water treatment

With the rapid development of the world economy, environmental pollution has become more and more serious, especially for water resource [98–100]. The pollutants in wastewater include heavy metals, organic dyes and pathogens [101–103]. Various disinfection technologies have been developed to remove pathogen bacteria in drinking water, including free chlorine, ozone and N-halamine. However, these disinfectants suffer from non-reusability and instability. Unlike these traditional disinfectants, Ag/AgX nanostructures have attracted extensive attention in water treatment due to their high yield, low cost, low energy consumption, and environmental friendliness [104, 105]. For example, Zhou et al. [106] deposited Ag–AgCl composite on $BiVO_4$ (Ag–AgCl/$BiVO_4$) via calcination to improve the photocatalytic effect under VL irradiation for water bacterial decontamination. Xia et al. designed and prepared polyacrylonitrile (PAN)/AgBr/Ag fibrous membrane by combining electrospinning and wet chemistry (Fig. 18) [107]. The fibrous membrane in the water had good photocatalysis, sterilization, and filtration properties. In addition, Lv et al. [108] reported a one-pot synthesis approach of CNC-Ag@AgCl with antifouling and antibacterial properties for water treatment. In Cheikhrouhou et al.'s report [33], a hybrid Cotton-AgCl was prepared and found to be efficient antimicrobial agents for water disinfection under sunlight irradiation. Moreover, an environmentally friendly Ag/AgCl functional cotton fabric for water disinfection and purification was, respectively, prepared [109–111].

5.3 Other applications

In addition to wound healing and water treatment, Ag/AgCl nanostructures have been employed in other fields, including food packaging, antioxidant, anticancer, etc.

Table 1 Summary of antibacterial behaviors of Ag/AgX-based composite materials

Entry	Nanomaterial	Antibacterial mechanism	Conditions	Bacteria	Refs.
1	Ag/AgX-CNTs (X = Cl, Br, I)	RSs[a]	435 nm, 20 min	*E. coli* K-12	[7]
2	Ag/AgCl/rGO	ROS[b]	–	*E. coli, S. aureus*	[15]
3	Ag@AgCl nanocubes	RCS[c]	532 nm, 10 min	MRSA, *E. coli*	[16]
4	Ag/AgCl/$W_{18}O_{49}$ nanorods	ROS	Full spectrum light	*Vibrio natriegens*	[17]
5	Ag/Ag/$CoFe_2O_4$	ROS	VL, 30 min	*E. coli*	[20]
6	Ag/AgX (X = Cl, I)/cellulosic fibers	ROS	–	*E. coli, S. aureus, C. albican*	[24]
7	Ag/AgCl/ABBN	ROS, Ag^+	–	*E. coli*	[25]
8	Ag/AgBr/MSNs	ROS, Ag^+	Solar light	*E. coli, S. aureus*	[31]
9	Ag/Ag@AgCl/ZnO	Ag^+, Zn^{2+}, ROS	VL	*E. coli, S. aureus*	[32]
10	AgCl/Ag	ROS, Ag^+	15 min	*E. coli, S. aureus*	[33]
11	Ag-AgX/RGOs (X: Cl, Br, I)	RSs, Ag^+	VLD, 8 min	*E. coli* K-12	[35]
12	Ag/AgBr/g-C_3N_4	ROS	VL, 120 min	*E. coli*	[36]
13	Ag/AgBr/TiO_2	ROS	VL ($\lambda > 420$ nm)	*E. coli*	[37]
14	Ag@AgCl/PAM-PDA hydrogel	ROS, PTT[d]	VLD, 10 min	*E. coli, S. aureus*	[39]
15	Ag/AgI/TiO_2	Ag, AgI, ROS	VL	*E. coli*	[40]
16	Ag@AgCl/ZnO	ROS, Zn^{2+}, Ag^+	VL	*E. coli, S. aureus*	[41]
17	Ag–AgCl NPs	ROS, Ag	VL	*L. monocytogen, S. aureus, S. saprophyticus, E. coli, P. putida*	[42]
18	Ag@AgCl	ROS, AgNPs	–	*S. aureus, L. monocytogenes* 1298, *P. MU2e*	[43]
19	AgCl/Ag-cellulose	ROS, AgNPs	–	*E. coli, S. aureus*	[44]
20	AgCl@Ag@TiO_2	$\cdot O^{2-}$, $\cdot Cl^0$	VL ($\lambda \geq 420$ nm)	*E. coli* K12	[46]
21	Ag/AgBr@Fe_2O_3	ROS	VL, 13 min	*E. coli*	[56]
22	GO-AgCl/Ag		VL	*E. coli, S. aureus*	[64]
23	Ag/AgCl/$ZnFe_2O_4$	ROS	VL, 15 min	*E. coli*	[76]
24	1-D core-shell Fe-Ag@AgCl	ROS	VL, 120 min	*E. coli*	[78]
25	C−CoFe/Ag−AgX (X = Cl, Br, I)	ROSs	VL, 40 min	*E. coli*	[79]
26	Double-shell Ag/AgCl/G-$ZnFe_2O_4$ nanocubes	ROS	$\lambda > 420$ nm	*E. coli, S. aureus*	[94]
27	Ag/AgCl	ROS	–	*E. coli*	[95]
28	H-PPAN/rGO-g-PAO@Ag^+/Ag	ROS	UV light	*E. coli, S. aureus*	[100]
29	PAN/AgBr/Ag	Ag^+	UV light	*E. coli*	[107]
30	CNC-Ag@AgCl	Ag^+	VL	*E. coli; S. aureus*	[108]
31	AgCl@SiO_2	Ag^+	VL	*E. coli*	[112]
32	Ag–AgCl NPs	Ag^+	–	*E. coli, B. subtilis, P. aeruginosa, S. aureus*	[113]
33	Cellulose/AgCl/Ag hybrids	–	–	*E. coli, S. aureus*	[114]
34	Ag/AgCl NPs	Ag^+, Cl^-	–	*S. aureus, E. coli, P. aeruginosa, B. cereus*	[115]

[a](Reactive species)

[b](Reactive oxygen species)

[c](Reactive chlorine species)

[d](Photothermal therapy)

[42–44, 112–115]. For instance, in Yu et al.'s report [41], the pectin (CEP) was employed to prepare Ag@AgCl/ZnO. The products worked as an efficient VL catalytic antibacterial agent, which could be applied in the fields of food packaging and sterilization.

6 Challenges and prospects for Ag/AgX nanostructures

Although the Ag/AgX antibacterial agents have developed rapidly, the severe aggregation of Ag/AgX to some extent reduces the active surface area, resulting in the loss of photocatalytic activity [116]. Therefore, it is necessary to explore a feasible approach to synthesize uniformly distributed anti-aggregating Ag/AgX to maximize the photocatalytic efficiency. Several strategies have been explored for structure optimization to improve photoactivity of Ag/AgX photocatalysts, such as the self-assembly approach and structural design. Ag/AgX self-assembled structures [117], including cubic cages, coaxial three cubic structures and nanoframes, expand the active surface area to realize enhanced light scattering and absorption.

Besides, bactericidal function, stability, and biocompatibility of Ag/AgX nanostructures are also crucial for their practical applications. Unlike some antibiotics, Ag/AgX nanostructures counteract pathogen without causing antibiotic resistance. Also, increasing the content of Ag^0 and reducing the particle size could probably improve the antibacterial activity and stability of Ag/AgX nanostructures. Besides, there is always impetus for development of Ag/AgX nanostructures with low minimal inhibitory concentration (MIC) and minimum bacterium concentration (MBC), as high doses of Ag/AgX nanostructures will lead to the excess release of Ag^+ that may cause high cytotoxicity.

Table 1 summarizes the typical antibacterial actions of some Ag/AgX-based composite materials [7, 15–17, 20, 24, 25, 31–37, 39–44, 46, 56, 64, 76, 78, 79, 94, 95, 100, 107, 108, 112–115].

7 Summary and outlook

The environmentally friendly, visible light-responsive photocatalysts are gaining worldwide popularity due to their advantages over traditional antibiotics and offer opportunities in the antibacterial field. Serving as a class of silver-based photocatalysts, Ag/AgX exhibits powerful antibacterial activity against pathogenic bacteria under light conditions. This review summarized the recent advances on how to make use of Ag/AgX nanostructures as competitive antimicrobial agent, emphasizing their antibacterial mechanisms and practical applications. The influencing factors of Ag/AgX nanostructures on antibacterial activities were discussed. Some typical strategies were highlighted in this review to improve the antibacterial efficiency of Ag/AgX nanostructures, concentrating on morphology and size regulation, surface engineering, efficient use of sunlight, and the separation/transportation of the electron–hole pairs. However, there is still a lack of systematic research on Ag/AgX nanostructures, and an efficient and stable Ag/AgX heterojunction system needs to be explored for potent antibacterial fields.

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Declaration

Conflict of interests The authors declare that they have no conflict of interests.

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