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REVIEW ARTICLE

The mini-review for synthesis of core@Ag nanocomposite

Rui Wu [a,*], Fagen Zhang [b], Xiaohui Ji [a], Yang Liu [a], Xiaohua Guo [a], Gunghui Tian [a], Bo Liu [a]

[a] *Shaanxi Key Laboratory of Catalysis, College of Chemical and Environment Science, Shaanxi University of Technology, Hanzhong, Shaanxi 723001, China*
[b] *School of Environmental Science and Engineering, Guangzhou University, Guangzhou 510006, China*

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Abstract As common noble metal, Ag nanoparticles (Ag NPs) have unique physical and chemical properties and it is widely used in daily life, scientific research, industrial production, environment, medical and healthcare systems. Core/shell of Ag nanocomposite have attracted attention due to the significant properties and wide applications by combination with the core and shell. As a mini-review, it is emphasized the approaches for fabrication Ag shell coating on core nanoparticles as core@Ag nanocomposite, including situ growth method, phenol formaldehyde resin method, silane coupling agent method, ultrasound method, PEI method, dopamine method and other methods. The procedures and mechanism for construction of core@Ag nanocomposite are highlighted. The comparison of those approaches, including their advantages and disadvantages are described in detail. At the same time, the properties and applications of Ag NPs are briefly introduced. The current challenges and perspectives in the future for synthesis of core/shell nanocomposite is proposed. The review is very useful for quickly gaining knowledge and experience for synthesis of core@Ag nanocomposite.

Contents

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[*] Corresponding author.
E-mail address: hftffc@163.com (R. Wu).

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1. Introduction

Materials, information and energy are the essence for human being towards civilization and social advance. Materials are the basic matter for people survival and development. The past two decades have witnessed the rapid advance of nanomaterials. As an integrated research field, nanotechnology aim at investigating and controlling nanoscale materials from chemistry, materials, biology, engineering, medicine, and so on. Nanotechnology is driven by the desire to discover novel, unique, smart, intelligent and advanced materials (Gong et al., 2020; Reimhult et al., 2019; Khan et al., 2017; Hartshorn et al., 2018). The advances in new nanotechnology make it possible to synthesize not only the spherical shape nanoparticles but also a variety of shapes such as rod, tube, cube, prism, disk, wire, polyhedron, core/shell, etc. It is worth noting that core/shell nanoparticles are also highly achievable as reported in articles (Chaudhuri et al., 2012; Hu et al., 2014; Su et al., 2020; Liu et al., 2021). Compared with single-component nanomaterials, core/shell composites have many advantages. Core/shell nanomaterials combine the properties of core and shell. In addition, there is also a synergistic effect between core and shell. At the same time, the thickness of core and shell can be tuned, thus endow them novel properties. The shell can prevent the aggregation of the core and protect the core. For some unstable and harmful substances, the shell can prevent leakage of the core.

For nanotechnology, a great number of materials such as noble metal nanomaterials (Ag, Au, Pt, Pd), rare earth fluoride ($NaYF_4$, $NaGdF_4$, LaF_3), quantum dot, graphene, carbon nanotube, magnetic nanoparticles (Fe_3O_4), hydroxyapatite nanoparticles are popular with researches (Lv et al., 2019; Wang et al., 2021; Zhu et al., 2018; Sun et al., 2018; Wu et al., 2019; Fatimah et al., 2021; She et al., 2021; Zhang et al., 2016). Compared with their bulk counterparts, nanotechnology endow core/shell of Ag NPs with distinct advantage. Ag NPs is an excellent nanomaterial with fascinating properties. As a mainstay of science and technology, Ag has been emerged for thousands of years. So, combine the core/shell structure, the core@Ag NPs display versatile advantages over simple nanoparticles. Verma fabricated a series of core/shell of Ag NPs for optical, electronics and sensing application. (Bhatia et al., 2021; Bhatia et al., 2020; Bhardwaj et al., 2019; Sekhon., et al., 2012; Bansal et al., 2016; Sekhon, et al., 2013). Herein, the approaches for synthesis of core/shell of Ag nanomaterials is discussed. Strictly speaking, the approach for Ag coating on core nanoparticles as shell to form core/shell is reviewed.

2. The properties and applications of Ag nanoparticles

As noble metal material, Ag NPs present many special properties and significant applications in many fields. It has favorable biocompatibility, optical and electrochemical properties, as well as antibacterial activity. So it is widely used in optics, catalyze, antibacterial, chemical detection, biological sensing and biomedical diagnosis (Shanthil et al., 2017; Sawant et al., 2018; Li et al., 2017). In ancient times, silverware was frequently used in daily life because of antibacterial property. In recent years, due to the development of nanotechnology, Ag NPs have significant antibacterial properties compared with silver in bulk. It has significant inhibitory effect on gram bacteria. The antibacterial effect is related to the particle size and morphology of Ag NPs. Different sizes and morphology of Ag NPs perform different colors and antibacterial properties. Also, by incorporating of other nanomaterials such as TiO_2, MoS, Au, CuO NPs, the composite show versatile properties. Zhao synthesized composite Ag NPs by codope Mg and Ti to study the osteogenic and antibacterial properties in vitro (Zhao et al., 2015). Ag NPs have also been found to be capable of healing wounds and repairing cells. It is recognized as a non-toxic, biocompatible, natural antibacterial agent. So, in some cases, in order to reduce the toxicity of nanocomposite, Ag NPs is often used for coating on the core nanoparticles as protective shell to prevent leakage and form core/shell nanoparticles.

Ag NPs are considered as unique optimal Raman substrate and it is widely employed for Surface-Enhanced Raman scattering (SERS) because it can remarkable enhance Raman signals to detect molecular with high sensitivity due to optical properties (Gong et al., 2020). Compare with other substrates, the Ag NPs have the best enhancement effect due to surface plasmon resonance (SPR) and the rough surfaces greatly strengthen the local electric-fifield near the surface. When the probe molecules located on the surface of Ag NPs as “hot-spots”, their weak Raman signals can be tremendously

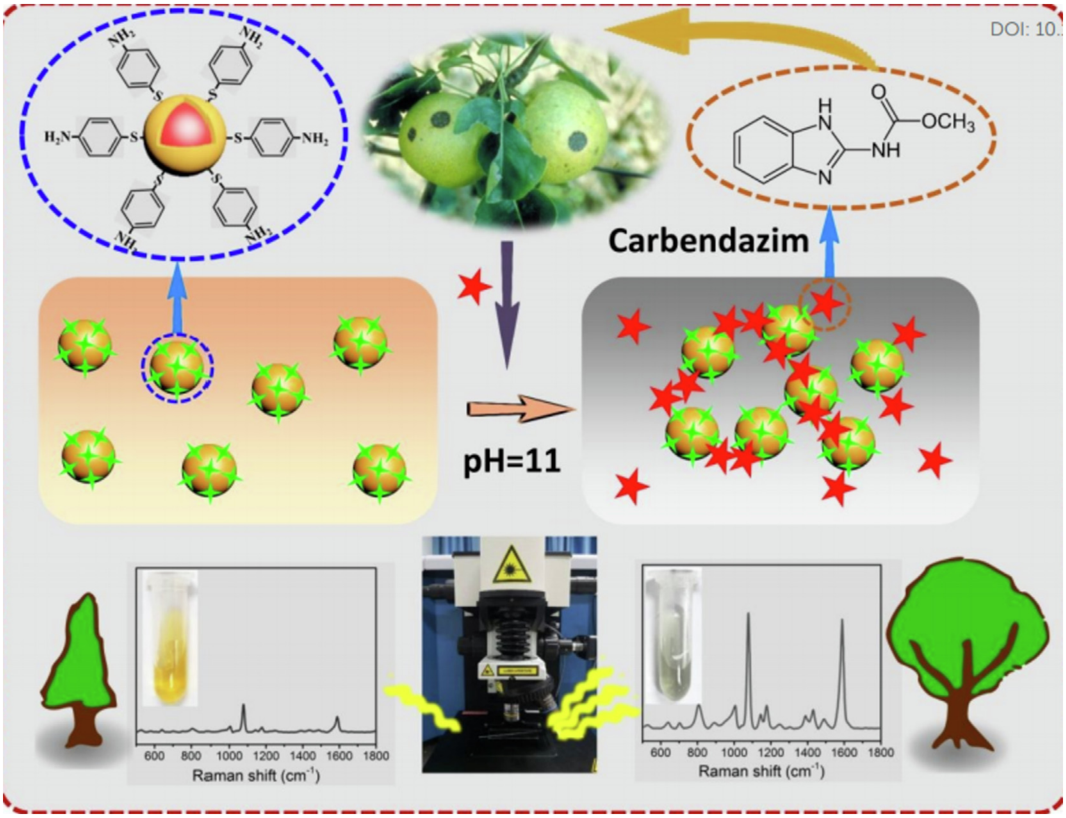

Fig. 1 Au@Ag Nanoparticle for Carbendazim detect.

enhance by the amplification of electromagnetic fields. The amplified signals endow with high sensitivity for specific analytes at trace amount. The mechanism can be explained in terms of electric field and polarization. SERS with Ag NPs is widely applied to detect ultra-low concentrations of pesticide residue in food, such as fruits, vegetables and grain. Wang design bimetallic Au@Ag nanoparticles for rapid sensing of carbendazim in fruit (Wang et al., 2020), as shown in (Fig. 1). The proposed method showed high sensitivity to carbendazim with a low limit of detection (LOD). In addition, it was applied for glucose detection (He et al., 2012), magneto-optical enhancement (Ortega et al., 2018;), and just as a protective shell (Michaud et al., 2019).

3. The approaches for synthesis of core@Ag NPs

The approach for Ag coating on core nanoparticles is based on synthesis of Ag NPs. The method for preparing Ag NPs is simple, but it is difficult to obtain Ag coating on core nanoparticles as shell (core@Ag) to form core/shell nanocomposite. There are many methods for synthesis of Ag NPs, such as physical, chemical and biological method (Khodashenas et al., 2015). The chemical methods include oxidation–reduction method, sol–gel method, hydrothermal and microemulsion method. Among them, the most common used method is oxidation–reduction method. Silver nitrate and Tollens' reagent act as raw materials. Common reducing agents are ascorbic acid, sodium borohydride, gallic acid, tannic acid, hydroxylamine hydrochloride, glucose, amino acids and polyethylene imine and so on.

3.1. *In situ growth method*

The fabrication of core/shell of Ag nanocomposites by situ growth method mainly refers to the Ag shell direct growth on some core nanoparticles. For some noble metals, such as Au, Ag, Pd and Pt, they have similar crystal structure and ion radius(Gong et al., 2013; Siddiq et al., 2019; Shrivas et al., 2020). When these noble metals nanoparticles act as core, the Ag NPs facilely anchored on the surface and gradually generate Ag shell. This seed-mediated growth not only applies for Ag coating on noble metals, but also applies for noble metals coating on Ag NPs. First, the noble metal nanoparticles are synthesized as core and washed with deionized water. Then, the noble metal nanoparticles are dispersed in water as seeds with stirring. Silver nitrate is added with vigorously stirring for several minutes. Mild reducing agent is added dropwise, slowly. Due to the close radius and the matched crystal lattice, the new produced Ag NPs directly deposite on the surface of the noble metal nanoparticles and generate Ag shell. It is important that the concentration of the silver nitrate and reducing agent should not too high with mild reducibility. Otherwise, it is feasible to nucleate spontaneously.

Jieli Lyu synthesized and characterized a series of Au@Ag nanoparticles with accurately measurable shape, structure and concentration of nanoparticles. The obtained thickness of the shell, overall size, polydispersity and number particle concentration by the various methods are in full agreement (Lyu et al., 2020). Tsuji designed Au@Ag core/shell nanocrystals via two-step reduction with organic solvents and discussed

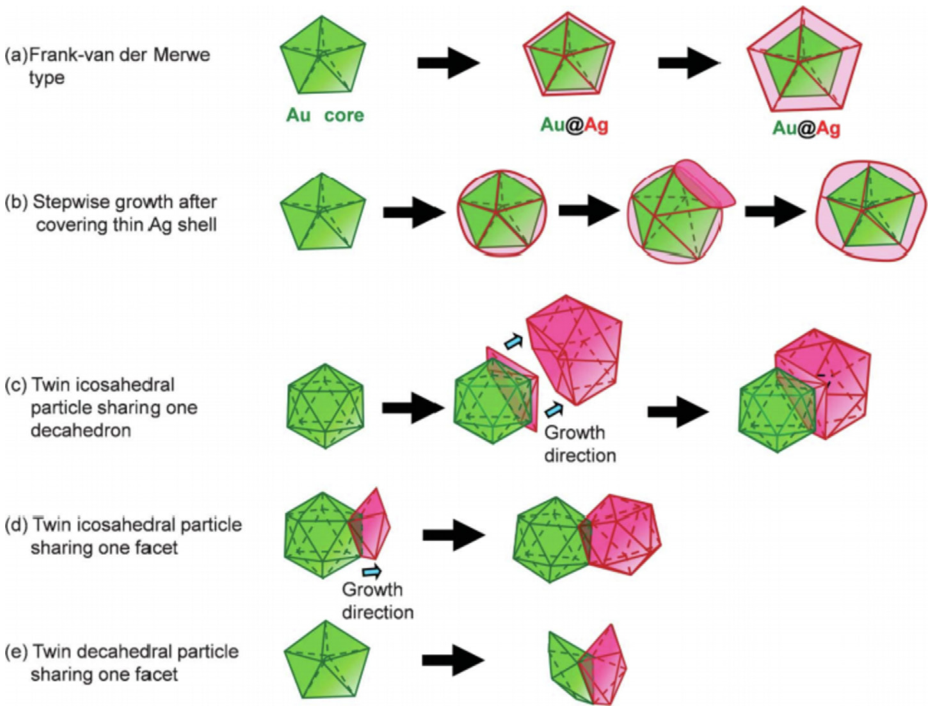

Fig. 2 Crystal structures and growth mechanisms of decahedral and icosahedral, Au@Ag nanocrystals and their twin crystals.

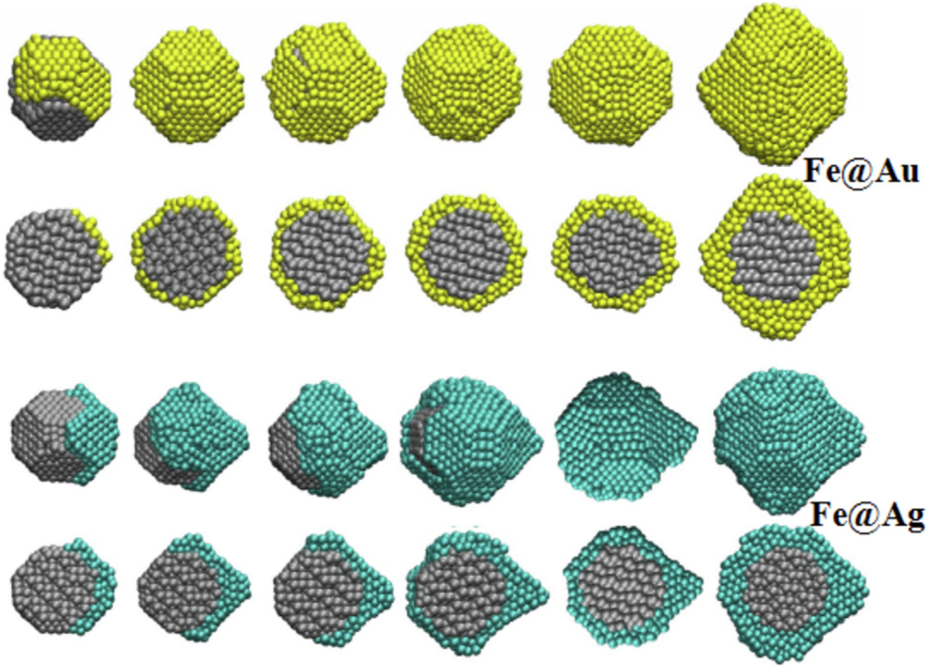

Fig. 3 Selected configurations of the Fe-Ag nanoparticles.

the growth mechanisms in detail, as shown in (Fig. 2) (Tsuji et al., 2012). In Fig. 2, it demonstrates the crystal structures and growth mechanisms of decahedral and icosahedral of Au@Ag. The thickness of the shell can be adjusted and the nanocomposite has novel SPR properties. Ségolène Combettes synthesized Fe@Au and Fe@Ag nanoparticles (Combettesa et al., 2020). Interface properties between Fe and the noble metals Au, Ag are computed by using discrete Fouriertransform(DFT) in detail, as shown in (Fig. 3), the whole process for Ag and Au nanoparticles gradually wrapping the Fe nanoparticles. Akbarzadeh studied Au@void@Ag yolk – shell nanoclusters by molecular dynamics simulation in order to study the effects of core and shell sizes on their thermodynamic stability and structural transformation at different

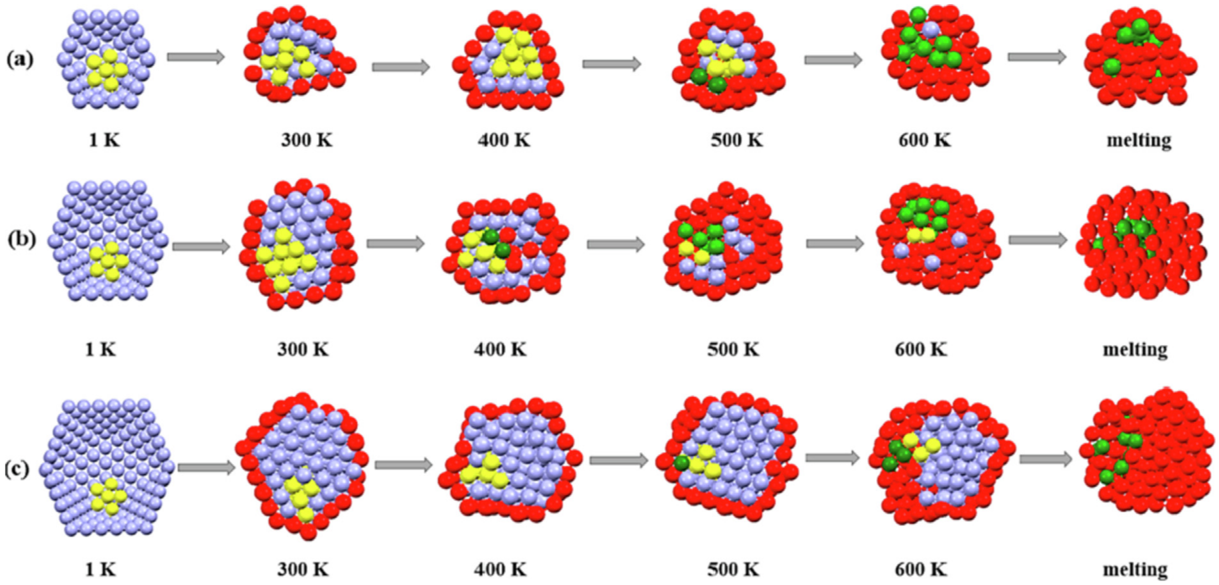

Fig. 4 Snapshots of Au@void@Ag at different temperatures during the heating process. Coloring denotes the type of atom: violet, solid Ag atom; yellow, solid Au atom; red, liquid Ag atom; and green, liquid Au atom.

temperture (Akbarzadeh et al., 2017). The effects of temperature on structure as shown in (Fig. 4).

3.2. PEI method

Polyethylenimine (PEI) is macromolecular polymer containing amino groups, with positive charge. The molecular weight ranges from hundreds to tens of thousands, and it's state changes gradually from liquid to viscous, filamentous liquid. PEI is often used as dispersant for synthesis of nanomaterials. When using PEI as bridge for coating Ag NPs, PEI can be added in the process of synthesis of core nanoparticles, or it can be added to the solution of the obtained nanoparticles, with vigorously stir or disperse by ultrasound (Zhou et al., 2013; Ansari et al., 2020). PEI can be uniformly adsorbed on the core and the free PEI is washed by deionized water. Then, small sizes of synthesized Ag NPs is added and stir. PEI contains a large number of amino groups, which have strong complexation with Ag. Due to the strong complexation, a large number of Ag NPs are adsorbed on the core. In order to get a layer of Ag shell, low concentration of silver nitrate can be added and then mild reducing agent such as hydroxylamine hydrochloride is dropwise added. In some cases, PEI can serves as reductant, but it needs to be heated at high temperature.

3.3. Dopamine method

Dopamine is a common biochemical reagent. Due to the adhesiveness and the large amount of amino group, it is often employed to conjugate other functional groups. For construction Ag shell, dopamine is dissolved in trius buffer solution, firstly. The buffer solution comprise trihydroxymethyl aminomethane and hydrochloric acid, the pH value is 8.5. The synthesized core nanoparticles are mixed with dopamine-containing of trius buffer solution, and they react for a certain minutes under ultrasound. So, polydopamine obtained under ultrasound, and coat on the core. Then, small size of Ag NPs are prepared via the reaction between sodium borohydride and silver nitrate. Finally, the small Ag NPs are added to the core with PDA. The core nanoparticles are covered with Ag NPs by the interaction between amidogen and silver (Fei et al., 2008; Chen et al., 2017). Follow this method, Keling designed Ag@HAp with PDA and study their properties, as show in (Fig. 5).

3.4. Silane coupling agent method

The molecular structure of silane coupling agent is generally Y-R-Si(OR)$_3$. Y and SiOR- represents organic functional group and silane oxygen group, respectively. Silane is liable to react with inorganic agents, and organic functional group is likely to react with organic agents. Therefore, the silane coupling agent act as binding layer between inorganic and organic interface. When the silane coupling agent is used for producing core/shell nanoparticles, the R group hydrolyzes to produce the silanol group, and then reacts with the hydroxyl group on the surface of the inorganic particle to form the hydrogen bond, and then form covalent bond. In addition, because of Y group with amine, it is easy to interact with Ag. When take this method for practice, the nanoparticles and coupling agent reflux in high temperature for a certain period to endow the core with amine group, and then cooled to room temperature. The free coupling agent wash away alternately by water and ethanol. Then, the core nanoparticles coated by coupling agent disperse in water by ultrasound. The prepared small Ag NPs mixed with the core for several minutes and deposited on the core. In order to obtain compact Ag shell, silver nitrate and mild reducing agent are added to the solution. The commonly used silane coupling agent is aminopropyltriethoxy silane (KH-550), namely APS (Wu et al., 2019; Zhou et al., 2017).

Sometimes, the SiO_2 first coat on the core by hydrolysis of tetraethoxysilane (TEOS) to form SiO_2 shell, then add silane coupling agent, the next process follow above. In other cases,

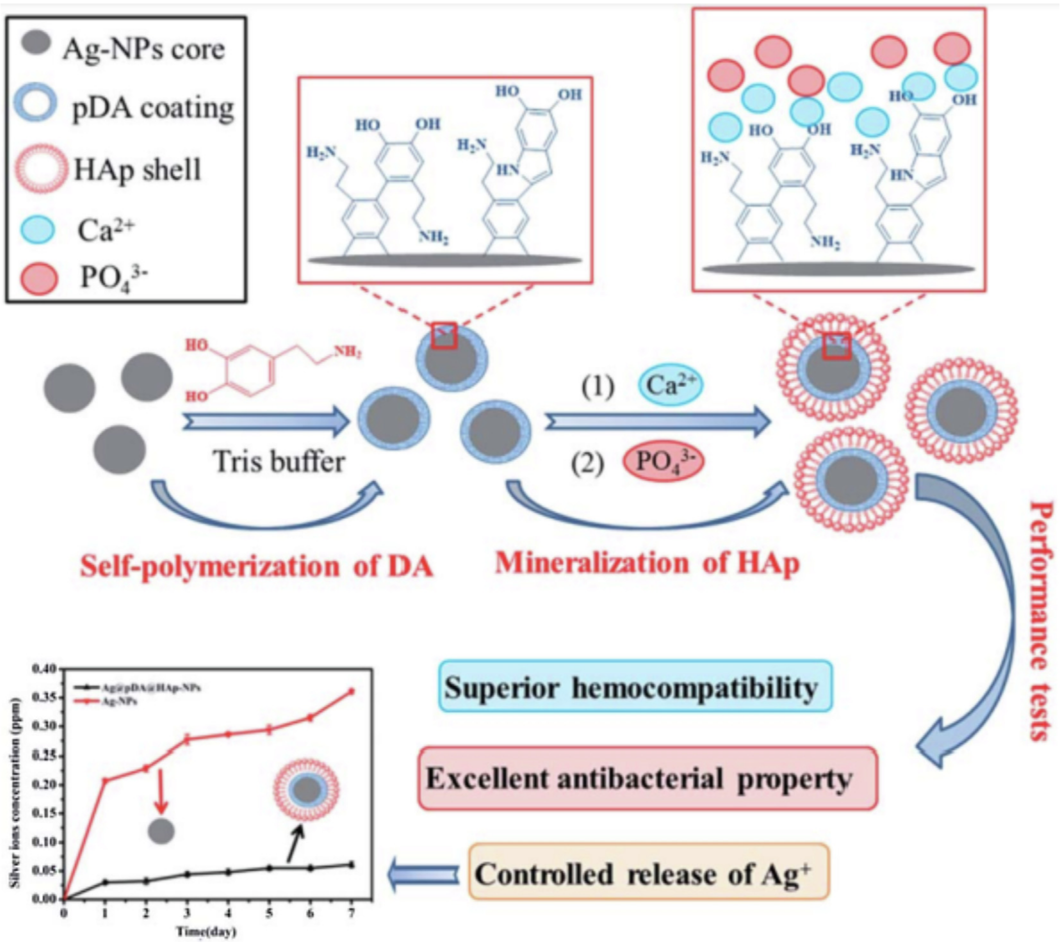

Fig. 5 The synthesis of Ag@HAp with PDA and property study.

after coating SiO_2 shell, the formation of Ag shell depend on ultrasound. First, the deionized water produce $H^\cdot$ and $OH^\cdot$ free radicals under ultrasound. The radicals neither return to their original state nor produce H_2 and H_2O_2. At the same time, under the attack of ultrasound, ethanol generates CH_3. and CH_2OH. In alkaline condition, $AgNO_3$ transform into $[Ag(NH_3)_2]^+$. Then, the positive charge of $[Ag(NH_3)_2]^+$ adsorb on the surface of SiO_2 with negative charge. The CH_3. and CH_2OH. react with $[Ag(NH_3)_2]^+$ to produce Ag and the Ag shell grow on the surface of SiO_2 under ultrasound. By this method, Mohamed Abbas designed Fe_3O_4@SiO_2@Ag and SiO_2@Ag nanoparticles (Abbas et al., 2015; Panigrahi et al., 2013), and studied their superior catalytic reduction for 4-nitroaniline, as shown in (Fig. 6). They fabricated SiO_2 and Fe_3O_4@SiO_2 by ultrasound and then bind Ag NPs under the help of ultrasound again.

3.5. Phenol formaldehyde resin method

Phenol-formaldehyde resin is a kind of phenolic resin obtained by condensation of phenol and formaldehyde under heating condition. It usually acts as adhesive because of high viscosity. When the resin is employed to design Ag shell, a layer of phenol–formaldehyde resin coat on the core (Cao et al., 2020). First, the synthesized core disperse in anhydrous ethanol under ultrasound and add ammonia with stir. Then, phenol and formaldehyde as raw materials are added and react for some time under heating condition to produce resin. So, the phenolic resin coat on the core. Wash away the free resin and the core disperse in water. After that, silver nitrate is added. Because of the resin with negative charge, and the silver ions with positive charge, the opposite charges attract each other which lead to the silver ions adsorb on the core. Silver nitrate is reduced to Ag NPs on the surface of the core by radiation of xenon lamp. Eventually, the Ag NPs coat on the core, the process is shown in (Fig. 7).

3.6. Thiols method

There is strong covalent interactions between thiol and metals which can regulate the morphology and endow new functions for nanostructure (Chen, et al., 2018;). Alkanethiols and aromatic thiols with functional groups, such as hydroxyl and carboxyl groups can accelerate the Ag deposition. It is demonstrate that the aromatic thiols, benzene ring can enhance the electron donating capability and thus boost their interactions with Ag^+ by theoretical calculations. Jiaqi Chen synthesized Au@Ag by aromatic thiols (Chen et al., 2019),

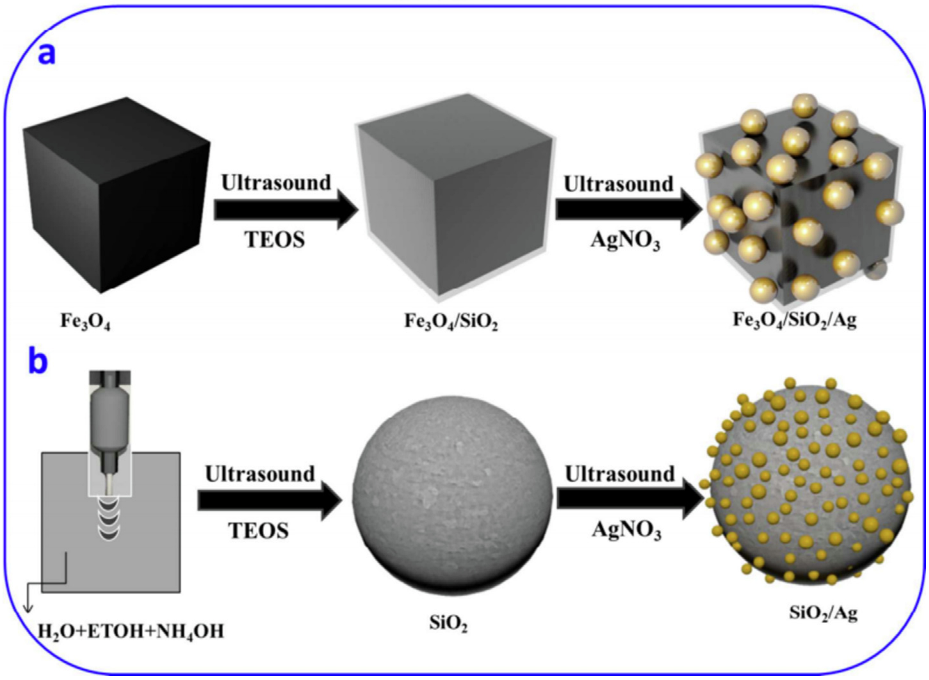

Fig. 6 Schematic diagram illustrating the synthesis process of (a) the silver-decorated silica-coated iron oxide ($Fe_3O_4/SiO_2/Ag$) nanocubes and (b) the silver-decorated silica (SiO_2/Ag).

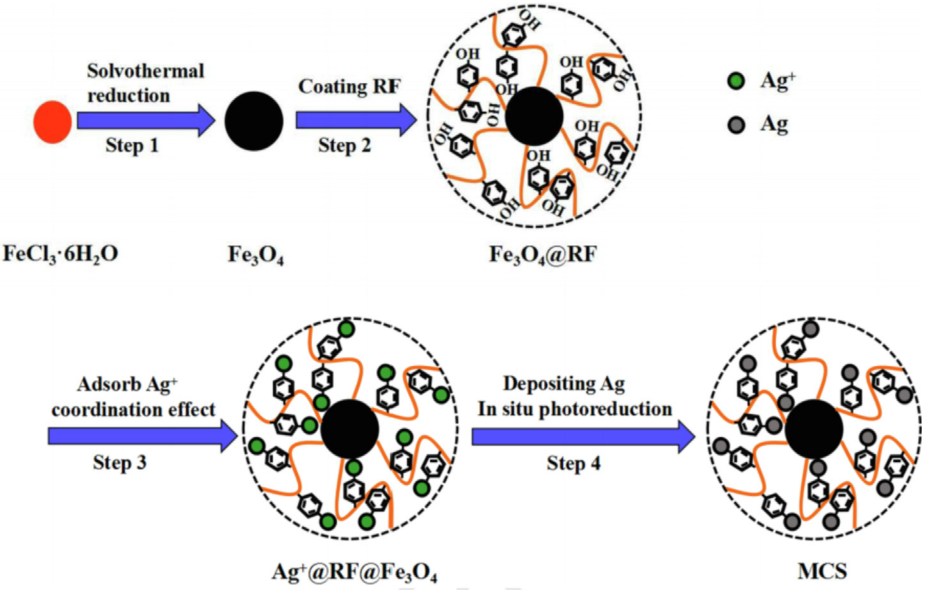

Fig. 7 Prepration of core/shell nanoparticles of $Fe_3O_4@RF@Ag$.

and they tracking thiol's position in the core/shell NPs for studying the mechanism. In addition, Qiaofeng Yao studied the thiolate-protected core/shell of noble metal nanoclusters: synthesis, self-assembly, and applications in detail, especially the molecular interactions/reactions (Yao et al., 2021). Puspanjali Sahu exploited dodecanethiol as bridge to fabricate Ag shell. (Sahu et al., 2015). Xiao Wei take thiol as bridge to synthesis Au–Ag Alloy nanocluster, as shown in Fig. 8 (Wei et al., 2021).

3.7. Other method

In addition to the above methods, for preparation of Ag shell, sodium citrate is a common bridge (Lyon et al., 2004; Ko et al., 2014; Li et al., 2016). It usually acts as reducing agent and dispersant. Sodium citrate is first added as a dispersant to synthesize core nanoparticles. Since sodium citrate has three carboxyl groups, on one hand, the carboxyl group chelate with the metal of core, making sodium citrate adsorb on core. On the

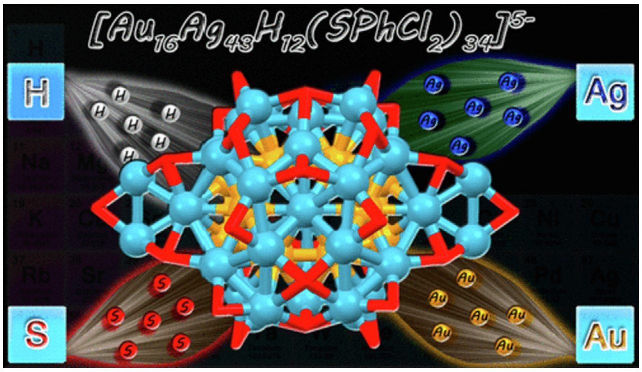

Fig. 8 The struct of $Au_{16}Ag_{43}H_{12}(SPhCl_2)_{34}]_5^-$.

other hand, because of the three carboxyl groups with negative charge, they repel each other. So the nanoparticles avoid aggregation and disperse well. Then the excess free sodium citrate is washed. The core with adsorbed sodium citrate are re-dispersed in water. Then, silver nitrate is added and heated to boiling. Ag NPs directly generate on the surface of core through oxidation reduction reaction between sodium citrate and silver nitrate. The disadvantage of this method is that it is difficult to form a dense and uniform silver layer.

Furthermore, in some cases, gallic acid and glucose are employed to prepare core/shell Ag NPs. When used gallic acid as reductant, Tollens' reagent is used as raw material. Tollens' reagent first adsorb on the core, then react with gallic acid to form silver shell (Li et al., 2013). When used glucose, core nanoparticles react with glucose via hydrothermal reaction in high temperature and high pressure condition. So, the core capped carbon, and carbon interact with silver nitrate to synthesize Ag NPs. Furthermore, microemulsion method, microwave method and plasma method are exploited to design core/shell of Ag nanocomposite (Miyakawa et al., 2014; Liu et al., 2015).

4. The advantages and disadvantages of the methods with comparison

Each approach has its own advantages and disadvantages. The in situ growth method is suitable for Ag shell growth on noble metals such as Au, Pt, Pd, Ag. The advantage of this method is that it only need silver source and reducing agent, without additional reagents and lack of complicated experimental steps. Also, the method is also suitable for wrapping core of Ag NPs with a layer of noble NPs. The disadvantage is that the Ag NPs are easy to spontaneously nucleate. Therefore, in the process of forming Ag shells, it is necessary to select appropriate reducing agent and control the solution concentration, and make the shells grow slowly. The advantage of PEI method is based on the complexation between abundant amino of PEI and silver, and the Ag particles can be strong fixed on the core. The PEI has different state, such as liquid, viscous, even solide, due to the different molecular weight. It is difficult to quantified and add the sticky PET. Furthermore, as a uncommon reducing agent, PEI can react with silver nitrate under high temperature. As a kind of macromolecule polymer, PEI should not be added too much,or may aggravate the aggregation of nanoparticles.

Compared with PEI method, dopamine method is more complicated due to the involvement of trius buffer solution. As a necessary substance of body, dopamine is safer than PEI. The synthesized core@Ag by dopamine can be used in biomedicine. Maybe silane coupling agent method is an universal method. Silane coupling agents easily bind to a variety of inorganic nanoparticles. Then the Ag NPs are loaded by the complexation of amino. Ultrasound method is similar with the silane coupling agent method, they all use silane coupling agents. The difference is that the free radicals are produced by ultrasound and play a significant role. Phenol formaldehyde resin method mainly uses the mutual attraction of positive and negative charge to fix Ag. The amount of loaded Ag^+ is limited in this method and the shell is not very dense. In addition, the phenol and formaldehyde are toxic substance. Also, the thiols method have the same problem which the compounds with sulfhydryl groups are toxic with unpleasant smell.

Among these approaches, the in-situ growth method is the simplest, but it is limited to noble metals for nuclear materials. PEI method, dopamine method and silane coupling agent method are similar in that they all load Ag through the complexation between amino group and Ag. Some other approach such as polyaniline (PANI) is similar with PEI method.

5. Summary and outlook

The development of core/shell of noble metals nanoparticles attract significant attention due to the combination of their unique mechanical, optical, electrical, chemical, magnetic and other properties, especially the core/shell of Ag NPs. In the paper, the approaches for synthesis of core@ Ag NPs are summarized. The Ag coating core nanoparticles as shell is described in detail, which lays foundation for the application of core/shell of Ag composite nanomaterials. This work puts forward a prospective strategy to construct core/shell of Ag NPs, and shows great potential for applications. In order to obtain better properties and broaden its applications, the technology for synthesis of Ag shell need to be improved. The challenge is how to control the morphology and reduce aggregation, as well as mass production. The morphology of nanoparticles determine their properties and applications. The aggregation for nanomaterila is an unsolved problem for scientists all over the world. In addition, pharmacokinetic studies and toxicity of the core@Ag NPs in vivo and vitro are another problem. Through innovation in technology and the efforts of scientists, it will endow core/shell of Ag nanocomposite with more novel properties and extensive applications in the future. Combine with the excellent biological compatibility and feasible modification of Ag NPs, the core/shell of Ag NPs will widely used for SERS, photocatalysis, antibacterial study, biological detection, medical diagnosis, electrochemistry. Especially the SERS, the amplified Raman signal can significant improve the detection degree of sensitivity and accuracy for analytes in environment. With the rapid development of the advance nanotechnology, the core/shell of Ag NPs will be applied in more fields.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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We greatly appreciated the Key Scientific Research Program of Shaanxi Provincial Education Department (21JS004) and the Brainstorm Project on Social Development by the Department of Science and Technology of Shaanxi Province (2021SF-382). As a mini-review, the design strategies of core/shell of Ag nanocomposite was systematically summarized. It include in situ growth method, phenol formaldehyde resin method, ultrasound method, silane coupling agent method, PEI method, dopamine method and other methods. The representative process and mechanism for synthesis of core/shell of Ag nanocomposite was presented in detail. At the same, the properties, applications and the method for synthesis of Ag NPs was briefly described. The current challenges and perspectives on the future for synthesis of core/shell of Ag nanocomposite was proposed. The review pavy the way for quickly gaining knowledge and experience for synthesizing of core/shell of Ag NPs.

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