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Recent trends in manufacturing of silver nanoparticles and future applications

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Recently, there has been a sudden surge in anti-microbial or anti-bacterial materials, due to contagious viruses and bacteria. These bacteria spread exponentially by mutations and have developed immunity towards many antibiotics and other drugs over time. Hence, there is a need for an alternative mechanism to deactivate their severity and thus reduce their effects. In this regard, silver nanoparticles exhibit great potential in terms of anti-microbial properties. They deactivate the viruses and thus prevent their further mutation. This has led to a growing demand for silver nanoparticle-decorated materials, which ranges from clinical instruments to routine life materials. In this paper, we have focused on the manufacturing techniques involved in the manufacturing of AgNPs. These involve both top-down and bottom-up approaches such as physical methods, chemical methods, green synthesis, along with advanced techniques such as 3D printing and 4D printing. Considering the ample scope associated with silver nanoparticles, we have discussed the potential applications in consumer goods and industries which can be incorporated to prevent widespread infections.

Keywords: Silver nanoparticles; 3D printing; 4D printing; manufacturing.

INTRODUCTION

Nano-materials (1-100 nm materials) have gained great attention in the past few decades in many fields such as biomedicine, catalysis, energy storage, and sensors, due to their unique physicochemical properties as compared to their bulk states [1]. Silver nanoparticles (AgNPs) have received special attention, especially in the biomedical field due to their desirable properties such as high electrical conductivity, anti-microbial properties, optoelectric properties and so on.

Silver nanoparticles have unique optical, electrical, and thermal properties and are being incorporated into a wide range of products such as conductive inks due to their high electrical conductivity, stability, and low sintering temperatures [2]. Additional applications include anti-microbial fabrics, 3D printing filaments, optoelectronics. A few of the interesting applications include the use of silver nanoparticles for antimicrobial coatings, keyboards and wound dressings that continuously release a low level of silver ions to protect against bacteria. Due to rising concerns of hygiene and safety, due to the Covid-19 pandemic, AgNPs are looked upon as an important candidate to minimize the transmission of bacterial and viral infections.

Manufacturing of AgNPs

There are two broad categories of manufacturing processes of nano-materials:

1. Top-down approach
2. Bottom-up approach

In the top-down approach, bulk silver material is crushed down into fine particles by size reduction using numerous techniques such as grinding, milling, sputtering, and thermal/laser ablation. On the contrary, in the bottom-up approach, AgNPs are manufactured by using chemical, photochemical, and biological methods. The commonly used physical techniques involved are discharge, physical vapor deposition, high-energy ball milling, laser ablation, etc. The chemical techniques involve electrochemical, chemical vapor deposition, sonochemistry, sol-gel, co-precipitation, inverse micelles, or micro-emulsions, etc. Among the mentioned, wet chemical reduction techniques are extensively implemented for silver nanoparticles.

Recent inclination can be observed towards the green-synthesis of AgNPs using plant extracts as an alternative to the conventional synthetic methods. These greener syntheses of AgNPs offer a major upper hand over the conventional synthesis.

A) Physical vapor deposition

In this synthesis, AgNPs are produced by a vapor deposition technique, where a metallic silver precursor taken in a dish (also called boat) is vaporized, followed by the condensation of the vapor as carried by an inert gas. The vaporization is carried out in a tube furnace at 1 bar pressure and at a high temperature. In a study by McNally *et al.* [3], sputtered AgNPs were created alongside ethanol, a common solvent in laboratories. The procedure resulted in the creation of different-sized silver nanoparticles having varied morphology. The sputtering created a pink solution of AgNPs.

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Further, the relationship between methanol jet and AgNP size variation was studied.

Although the synthesis produces abrasion-resistant and impact-resistant AgNPs, it suffers from some drawbacks like high energy consumption, large space required for the furnace and slow rate of synthesis; hence it is expensive and not preferred for mass production.

B) Laser ablation

Another physical technique for the synthesis of AgNPs is laser ablation which produces colloidal AgNPs in a single step without any additional contamination, thereby eliminating the requirement of purification. It is cost-effective, safer and more environmentally friendly. Laser ablation system consists of a powerful laser source and feeding system. The laser beam is focused on the metal substrate which causes multiple metal particles to rise perpendicular to the substrate, commonly referred to as 'Plume'. These particles are then directed towards the collector using inert carrier gas. The concentration, size and distribution of AgNPs is influenced by the medium used in the ablation chamber, metal substrate used and laser operating parameters. Experimental estimations showed that the mass of generated nanoparticles in ambient air was up to 100 times higher than in water and that in argon gas was up to 100 times higher than in ambient air [4]. However, in terms of other preparation methods, laser ablation exhibits a few drawbacks as well. It incurs high investment costs because of the high price of the laser system. The process needs to be implemented on a large scale for being economical and sometimes there is a chance of particle agglomeration due to plume formation.

C) Electrochemical synthesis

In the electrochemical method, nanoparticles are produced by the dissolution of a metallic anode in a suitable solvent. For the synthesis of AgNPs, the silver anode is subjected to electric current. The particle size is controlled by administering the applied current density. As cited by Khaydarov *et al.* [5], inexpensive bulk silver electrodes were selected to produce AgNPs using electrochemical process. The electrodes were then placed in an electrochemical cell. The process was performed at 20–95 ^{o}C at a constant voltage of 20 V. The morphology of AgNPs produced by the proposed method was studied. Spherically shaped AgNPs were produced exhibiting stability of up to 7 years under ambient conditions. The physical technique permits the production of mass-scale AgNPs in a single-step process. Electrosynthesis has some disadvantages too. It usually requires the use of a solvent to stabilize the reactants and products. Water is the ideal solvent but often organic solvents or co-solvents are required. Additionally, supporting electrolytes to carry the current are very often needed.

D) Chemical approach

Chemical synthesis is the most preferred approach for the synthesis of AgNPs due to its high yield and low cost. The general route for manufacturing silver ions is reduction using reducing agents followed by their stabilization using stabilizing and capping agents. Fine size and distribution of silver nanoparticles are achieved by producing the nuclei at the same time from the supersaturated conditions, with subsequent growth and stabilization. The parameters used for controlling the size and distribution of Ag ions include the concentration of silver ions, the concentration and efficacy of the reducing agent, the concentration and nature of the stabilizing agent, along with the reaction temperature, pH, pressure, etc. [6]. Different shapes and sizes of AgNPs can be obtained by utilizing suitable conditions and a shape directing agent. Although a great variety of chemical methods are available, most of the chemicals are toxic and cause environmental pollution and the synthesized AgNPs become cytotoxic. As these are technically and economically unfavorable, alternative greener methods are preferred.

E) Photochemical approach

The photochemical approach is a comparatively greener technique to produce AgNPs utilizing light energy. Reduction of Ag ions to silver occurs in the presence of photo-active chemicals such as acetone with isopropanol, carbon dots, modified clay suspension, etc. It can also act as a stabilizer for the nanoparticles, under exposure of UV or solar light irradiation, thus producing finely dispersed Ag nanoparticles. The most important parameters of the photochemical process are the capping and the stabilizing agent since they affect the size and distribution of AgNPs [7].

F) Green synthesis of AgNPs

As cited by Awwad *et al.* [8], numerous methods have been studied for the synthesis of silver sulfide nanoparticles (Ag_2SNPs) namely a template-based method at room temperature and ambient pressure, water-in-CO_2 micro-emulsions [9], modified homogeneous precipitation route [10], sonochemical synthesis, multi-solvent thermal decomposition method [11], modified chemical bath deposition technique, combretum molle black wattle extracts [12], metal-reducing bacterium *Shewanella oneidensis* MR-1, chemical method [13], multi-solvent thermal decomposition method [14]. These methods have many disadvantages due to toxic chemicals used and waste products, which create a problem to the environment, also high energy consumption, difficulty of large-scale processes and wasteful purification. Recently, green synthesis has gained great attention due to its eco-friendly nature.

Herein, the objective of the present research work was to synthesize silver sulfide nanoparticles by a green route using rosemary (*Rosmarinus officinalis*) leaves aqueous extract at room temperature and studying antibacterial activity. Based on the paper, we cited that synthesized silver sulfide nanoparticles by the green synthesis method were studied for antimicrobial activity using the disc diffusion method; it was observed that silver sulfide nanoparticles have antibacterial activity at different concentrations. Chloramphenicol was used as a control antimicrobial agent. The Ag_2SNPs bio-synthesized by rosemary leaves aqueous extract displayed inhibition against gram-negative *Escherichia coli*, and gram-positive *Staphylococcus aureus*, *Shigella*, listeria bacteria. Thus green synthesis provides a cleaner route for the manufacturing of silver nanoparticles with considerable anti-microbial properties.

G) Methods and approaches for 3D printing with nanocomposites

Many processes have been explored for the manufacturing of AgNps. However, the possibility of manufacturing intricate and complex geometries with all materials (especially at nanoscale) is not an easy task. Newer approaches for the manufacturing of components with nano-composites will make it more accessible for printing a wide array of materials, as cited by Challagulla *et al.* [2].

H) 3D printed filaments

AgNPs in form of filaments can be effectively used for 3D printing (additive manufacturing) which provides extensive flexibility with regard to manufacturing. In a literature review of Podstawczyk *et al.* [15], we found that the filaments were produced by using dried PLA pellets dissolved in DCM with a concentration of 100 mg/mL under magnetic stirring with a rate of 300 rpm for 5 hours. The temperature was set at 35ºC to fully dissolve the polymer (solution A). The next step involved the preparation of silver nitrate solutions with concentrations ensuring an appropriate silver content in the final solid nanocomposite (0.01%, 0.1%, 1%, 2.5%, and 5% w/w). Subsequently, solutions A and B were mixed under stirring until a homogeneous mixture was obtained (about 30 minutes), which was transferred onto petri dishes and placed in a laboratory drying oven with the temperature set at 50 ºC for 48 hours to evaporate the solvents. Next, the dried material was mechanically ground using a blender and extruded by a screw extruder (Felfil Evo) with a nozzle of 1.75 mm at a temperature of 165 ºC to 170 ºC (temperature recommended by the extruder supplier). Filaments with diameters between 1.6 mm and 1.9 mm were chosen for 3D printing.

The antibacterial activity of 3D printed $AgNO_3$-PLA cuboids was evaluated against gram-positive *S aureus* sp *aureus* ATCC 6538, as well as gram-negative bacteria, *E. coli* ATCC 10536 and *Pseudomonas aeruginosa* ATCC 15442. Taking inspiration from this method, we can create filaments required for 3D printing which provides us with immense flexibility in terms of manufacturing low-volume complex materials. Thus, the antimicrobial activity of AgNPs coupled with advancements of 3D printing technology will enable new domains of manufacturing and applications.

I) Ink jetting of AgNPs

Material jetting is celebrated as one of the crucial enablers of MFAM (multifunctional additive manufacturing**)** because of its ability to print a wide range of materials including polymers, composites and inks containing metal nanoparticles. Inkjet printing (IJP) is a drop-on-demand, non-contact material jetting process capable of direct printing circuits of complex designs from a computer-aided design (CAD). It has advantages, namely, high material utilization rate and lower wastage compared to conventional methods. Ink-jet printing also has the potential to simplify the manufacturing process of circuits and reduce the associated costs.

In recent years, the use of inks containing MNPs such as silver, gold, copper and other conductive materials including graphene, carbon nanotubes, poly (3.4-ethylene dioxythiophene) polystyrene sulfonate and direct writing of liquid metals such as gallium, indium and tin have been extensively studied for printing circuitry. MNPs exhibit better resolution and thermal stability compared to conductive polymers and liquid metals and hence they are widely preferred for the manufacturing of circuitry.

In a study by Vaithilingam *et al.* [16], we cited that an ink containing AgNPs was printed using a bespoke 3D multi-material IJP system – JETx, followed by sintering using an in-built IR source. Essentially two sintering methods, swathe-by-swathe (SS) and layer-by-layer (LS) were employed to produce the AgNP ink. Briefly, during SS, each swathe of a layer was sintered using IR. It is envisioned that these sintering mechanisms can lead to variation in the layer formation, surface profile, micro-structure and the associated electrical resistivity of the printed circuitry. Currently, findings the on inkjet printing and sintering of AgNPs are limited to only a countable layer (less than 20) and are implemented in multiple steps (i.e. printing and sintering on different equipment). In the studied literature, 50 layers of silver were inkjet-printed and sintered in a single step and the impact of SS and LS mechanisms on the printed structure is reported.

J) Polymerization method (coating method)

There are multiple ways studied for the polymer and nano-silver composite incorporation, where AgNPs are dispersed on the surface of a polymer. Initially, a polymer is fabricated with AgNPs, with a subsequent attachment to the surface, a polymer solidification process occurs on the surface of the targeted object and then, it is incorporated with nano-silver particles. The monomer gets attached to the nanoparticle (NP) surface, followed by polymerization. In every case, the most crucial step is the attachment of the polymer to the surface of the object. We have focused on the manufacturing pathways of coating polymer nano-silver composites *via* various attachment procedures.

There are numerous methods established for the insertion of nano-silver into the polymer matrix. Nano-silver incorporation is predominantly performed *via* direct adsorption or *in-situ* synthesis. The first approach, which is the direct adsorption of nanosilver, is very controllable and straightforward; however, AgNPs can easily aggregate during this method. The following method, which is the *in-situ* synthesis, is comparatively harder because of the silver ion loading. However, it is more beneficial when compared to direct attachment adsorption due to the formation of homogeneously distributed silver nanoparticles in the matrices. This matrix demonstrates the benefits of inorganic materials, namely chemical stability along with mechanical strength, porosity which results in an ion exchange with the surrounding medium.

K) 4-D manufacturing

4D printing is useful for an array of healthcare applications, ranging from nanoparticle designing to tissue engineering, to the manufacture of self-assembling human-scale biomaterials. From the works of Ryan *et al.* [17], we cited that Organovo Holdings Inc., U.S.A., has been involved in several bioprinting projects focused on the development of functional human tissues. This company is implementing 4D manufacturing to develop an artificial human liver. One of the core techniques for 4D printing is to design the materials for structural change thus enabling a completely new domain of research and development. Even though the smart material itself plays a crucial role in transforming a printed object into another shape or configuration under the influence of external stimuli, attention should be given to understanding the mechanisms, predicted behaviors, and required parameters to achieve controllable results. The critical advantage of 3D printing technology is the ability to fabricate complex 3D shapes with varied material distributions through the spatial arrangement. By integrating the orientation and position of smart materials such as shape memory polymer fibres within composite materials, we can facilitate morphological changes in response to external stimuli. Incorporating anti-microbial materials such as AgNPs can further expand their applications for biomedical applications thus facilitating adoptions in the bio-medical field.

Since 4D printing and 3D printing are disjoint from conventional manufacturing technology, these new technologies can help reduce the manufacturing time and labor required to assemble machines or goods.

Extensive applications of silver nanoparticles

The unique characteristics of AgNPs make them and their nano-composites very useful in the fields of medicine, catalysis, textile, biotechnology, electronics, optics, water treatment, etc. Significant inhibitory effects against different pathogens display them as effective antimicrobial agents in various consumer products such as cosmetics, air sanitizer sprays, respirators, socks, slippers, pillows, wet wipes, toothpaste, washing machines, wound dressings, bone cement, surgical dressings, cell phones, and food storage packaging, etc. The use of heterogeneous AgNPs based catalysts is proven to be an effective and important technique in terms of efficiency and selectivity for different organic transformations, in conduction to their antimicrobial activity. Again, the cost of such catalysts is much less compared to corresponding gold or platinum (1/50) or even to palladium (1/25) catalysts [6].

Antimicrobial devices. AgNPs are extensively used in numerous biomedical devices, namely, surgical instruments, bio-sensing devices, vascular prostheses, orthopaedics, ventricular drainage catheters, wound dressings, heart valves, etc. Additionally, AgNPs are used for dental hygiene and eye treatment, as well as for other infections. Ointments and creams containing AgNPs are widely in use to avoid infections to the burns and to close the wounds. AgNPs are now also used as nanocarriers for a controlled drug delivery system.

Moving along the same lines, AgNPs can be extensively used in automobile interiors as an antimicrobial agent. The method commonly referred to as electro-spinning (or electrospraying, which yields nano- or microparticles due to lower solute concentration) is well-known and used in the production of nanomaterials, as well as for the theoretical study of electrohydrodynamic effects thus widening their applications. Electro-spinning's basic operation and nano-material production require a high-voltage power source (between approximately 50 and 500 kV/m) and two electrodes connected to opposite potentials. Thus, using the method of "electrospinning" we can produce AgNPs-impregnated fibres which can be used for automotive car interior (seat covers, dashboard, steering wheel cover and gear lever). A study by Mathavi *et al.* [18], indicates that an automotive steering wheel is the breeding ground for bacteria. A steering wheel is 11 times dirtier than a public toilet seat. A major source of bacteria comes from food spills, through air and heating vents and from the foot-wears of passengers. High concentrations of bacteria can be observed on dashboards, cup holders and children's car seats. The most common organisms include *Bacillus* species, *Staphylococcus*, *Escherichia coli*, *Salmonella* and *Campylobacter*. Thus, it is important to constantly clean and disinfect the car surfaces. Thus, using these anti-microbial materials helps to prevent the growth of micro-organisms and keeps the car safe and clean. This facilitates a significant reduction in viral infections thereby maintaining hygiene inside the vehicle.

As per the above study, the interior of cars was 8 times more infectious than a toilet seat. Incorporating AgNPs in seat covers, dashboards, gear level, handbrake lever can effectively mitigate the bacteria transmission to the passengers.

Opto-electronics. Silver nanomaterials have been extensively studied as an essential component of nanocomposites: thanks to their high dielectric constants in numerous systems. For example, silver nano-wires can be used as conductive coatings in flexible electronics and semiconductors due to their high electric conductivity. On the same lines, AgNPs have the potential to be utilized in silver paste for effective contact at electronic interfaces. Nanospheres consisting of Ag or Au have been utilized in optoelectronic light harvesting based on the plasmonic effect.

Silver-decorated composites. Black phosphorus (BP) has attractive nanoscale chemical and physical properties. In a study by Tang [19], black phosphorus decorated with silver nanoparticles (Ag/BP) was synthesized *via* an *in-situ* chemical reduction approach without utilizing a reductant.

The oil distributed with small amounts of Ag/BP nano additives exhibited excellent lubricating performance for the steel/steel contact surface. A significant reduction in coefficient was found. The friction reduction and wear reduction characteristics of Ag/BP are understood by studying the scar surfaces. During the operation, the Ag/BP nanomaterials served not only as a friction-reducing and anti-wear additive but also as a catalyst to decompose the base PAO oil for forming a carbon-based tribofilm further reducing the friction and wear.

The black phosphorus powder was prepared by high energy mechanical milling (HEMM). Briefly, red phosphorus and stainless-steel balls were put in a 50 ml stainless steel vial together with 10 mm, 6 mm and 4 mm milling balls After milling, the bulk black phosphorus was collected and finally stored in an argon glove box.

This technique can be essentially used for coating high-friction and wear surfaces such as piston rings. Piston rings are a highly dynamic and very important component from the point of view of I.C. engines. Incorporating low-friction coatings will not only improve the life of rings but also considerably improve the thermal performance. Further, the scope can be widened and effectively utilized to reduce frictional resistance in various machine components to improve the efficiency of the machinery and reduce wear.

Paint drying. We cited from the works of Lotfizadeh *et al.* [20] that the effects of nanoparticles on the paint-drying processes of automotive-based paints were experimentally investigated. In the study, rectangular aluminum plates covered by Alkyd Melamine (ES-665) car paint from Haviloox Company containing various amounts of 10 nm diameter silver nanoparticles from 0 to 25 ppm were prepared and tested. By varying the conditions such as air flow velocities, temperatures, and amounts of silver nanoparticles as important parameters during the drying process, an influence on the composition of the paint was indicated both during and at the end of the drying process, which has affected the quality of the final coating and improved the paint's chemical interactions. An increase of 22% at the surface temperature of the studied samples and the drying velocity was recorded for the 10 ppm nano silver amount, indicating an optimal nanoparticle amount, thus effectively reducing the time required for paint drying and corresponding lead times. This will help reduce the cost of manufacturing and improve the paint quality. Further, it can result in creating extensive room for development in the paint industry.

Covid-19 equipment. There is a sudden surge in the demand for anti-viral surgical masks due to the ongoing pandemic. AgNPs have again proved to be an ideal selection for the same. Impregnating AgNPs in masks can improve their anti-viral performance thus ensuring better protection against SARS Cov 2. As per the studies of Rasmi *et al.* [21], nano-disinfectant coating on surgical masks was extensively studied which indicated sufficient anti-viral abilities. The further scope can be improved by impregnating AgNP fibres into surgical masks. We can also incorporate AgNPs in PPE kits, gloves and other accessories predominantly utilized during Covid-19. Since AgNPs have proved to be effective when incorporated in gloves, we can further extend it and use it in other equipment.

CONCLUSION

Considering the widespread benefits and importance of silver nanoparticles, they've numerous applications both from consumer and industry points of view. As it can be observed, the applications range from day-to-day commodities and medical devices. In this paper, we have discussed the methodologies for the synthesis of silver nanoparticles. We have covered all the manufacturing techniques ranging from physical methods, chemical methods, green synthesis and 3D and 4D printing as well. Further, growing health concerns due to ongoing pandemic have created essential awareness among the people regarding hygiene and safety measures. This will boost the acceptance of anti-microbial devices which utilize silver nanomaterials. This will require the efficient and cost-effective synthesis of AgNPs and the development of new techniques.

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