

Dung dịch nano bạc, các phương pháp điều chế, những đặc tính và khả năng ứng dụng

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TÓM TẮT

Sự phát triển của vật liệu qua các thời kỳ: đồ đá, đồng, sắt, cao phân tử và hiện nay là vật liệu nano. Với các kích thước cực nhỏ, diện tích bề mặt rất lớn và hiệu ứng lượng tử vật liệu nano mang lại nhiều đặc tính vượt trội và ứng dụng đặc biệt. Vật liệu nano bạc (AgNPs) vừa mở rộng và bổ sung những đặc tính mới của Ag nên phạm vi ứng dụng cũng phát triển hơn, đặc biệt trong lĩnh vực môi trường, y học và bảo vệ sức khỏe con người. AgNPs được điều chế từ kim loại “trên xuống” hoặc từ ion “dưới lên” bằng các phương pháp vật lý, hóa học, hóa lý, sinh học hoặc kết hợp hỗn hợp. Sản phẩm AgNPs là dung dịch thật hệ keo có những đặc tính rất phụ thuộc vào các phương pháp điều chế, song những đặc tính cơ bản như cộng hưởng bề mặt plasmonic của hạt nano bạc bằng UV-Vis, hình dạng, kích thước và cấu trúc hạt bằng TEM, SEM, AFM, FTIR, XPS, XRD, phân bố cỡ hạt bằng Laser Scattering Particle Size Distribution Analyzer và Zeta Phoremeter Instrumentation. Nồng độ nano bạc được xác định bằng AAS, ICP-MS, ICP-OES. Tùy thuộc vào mục đích sử dụng vào lĩnh vực: xúc tác, quang điện, vi điện tử, môi trường, y dược, sức khỏe,... còn xác định thêm các phương pháp xác định các tính chất tương ứng. Do AgNPs có nhiều đặc tính đặc biệt nhất là lĩnh vực diệt nhiều vi khuẩn bảo vệ môi trường và sức khỏe con người nên chiến lược nghiên cứu phát triển AgNPs được đặc biệt chú ý tại nhiều quốc gia trên thế giới.

Từ khóa: AgNPs, các phương pháp điều chế, những đặc tính, khả năng ứng dụng.

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Silver nano solution: manufacturing methods, characteristics and applicability

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ABSTRACT

The development of human society is associated with the development of materials through the ages of stone, copper, iron, polymers and now nano materials. With extremely small sizes, very large surface areas and quantum effects of nanomaterials, nano materials offer many outstanding properties and opens up many special applications. Silver nanoparticles (AgNPs) have both the properties of metallic silver while expanding and adding new properties, so the application scope is also more developed, especially in the fields of environment, medicine and human health protection. Silver nano is prepared according to the principle of "top-down" from metal or "bottom-up" from ion by physical, chemical, physicochemical or biological techniques or a mixture of combinations. The obtained silver nano product is a true colloidal solution whose properties are very dependent on the preparation methods, but the basic properties are the nature of the plasmonic surface resonance of silver nanoparticles by UV-Vis, particle shape, size and structure by TEM, SEM, AFM, FTIR, XPS, XRD, nanoparticle and colloidal size distribution by Laser Scattering Particle Size Distribution Analyzer and Zeta Phoremeter Instrumentation. The concentration of nano silver is usually determined by methods such as AAS, ICP-MS, ICP-OES. Depending on the intended use in the fields of catalysis, photovoltaic, microelectronics, environment, medicine, health, etc., methods to determine the corresponding properties are also applied. AgNPs has many special characteristics, the most prominent of which is in the field of killing many bacteria and viruses to protect the environment and human health, so the AgNPs development research strategy is specially noticed in many countries in the worlds.

Keywords: *AgNPs, methods, characteristics, applicability.*

1. INTRODUCTION

Metallic silver was discovered thousands of years BC and has become a very precious metal used as currency in feudal society in many countries as well as jewelry and household items.¹ With properties as good conductor of electricity, heat, light sensitivity and antiseptic, silver has been used in the fields of electricity, electronics, film and medicine since very early. Since the

development of nanomaterials with effects on subatomic small size, large area and quantum,^{2,3} silver nanoparticles (AgPNs) have also been focused on researching innovations such as:^{4,5} electrical properties,⁶ electronic,⁷ catalytic,⁸ and especially antibacterial.⁹⁻¹¹ Because AgNPs have many applications in science, technology and life, especially with very good antibacterial ability,¹²⁻¹⁴ many research and manufacturing methods such as physics,¹⁵⁻¹⁷ biology,¹⁸⁻²⁰

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chemistry,²¹⁻²³ and electrochemistry²⁴⁻²⁷ have focused their research, including new method to create high purity or local green raw materials are available and cheap.

2. MANUFACTURING METHODS

2.1. Physical methods

2.1.1. The "top - down" approach

Fabrication of AgNPs by physical method follows the "top-down" principle with bulk metallic silver using a large amount of heat to separate the silver into vapor and then condense it like PVD,^{28,29} or granular and then dispersed. such as laser cutting^{30,31} or electric arc.^{32,33} Figure 1 shows the principle of laser method (a)³⁰ and arc discharge (b)³³ along with corresponding TEM images of the obtained AgNPs particle size and shape and size.

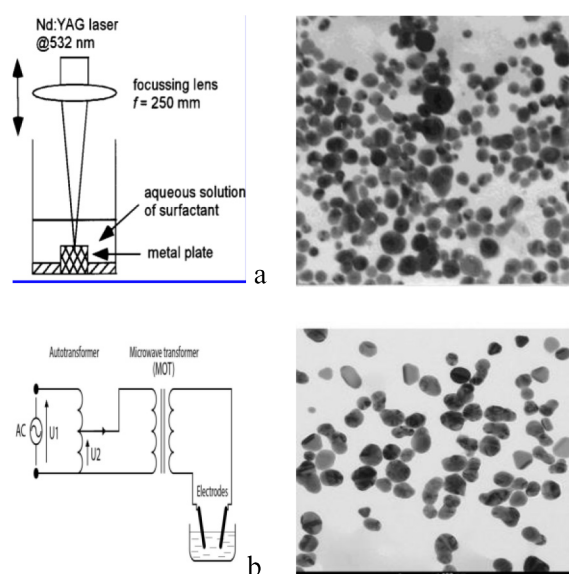


Figure 1. Schematic diagram and TEM image of AgNPs, a) Laser method,³⁰ b) arc discharge method.³³

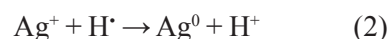
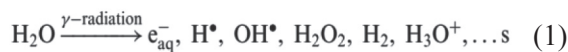
The AgNPs solution obtained by the above methods has a time-dependent light to dark yellow color and has a characteristic UV-Vis spectrum from 400 to 404 nm. Figure 1 shows that the shape of the nanoparticles is not uniform, so the particle size distribution spectrum is wide from 10 to 300 nm and the average is 46.8 to 48.9 nm. The zeta potential values from -20.4 to -22.31 mV show that AgNPs colloidal

solutions can be prepared by physical methods without the need for stable stabilizers. Although the production of AgNPs by the above physical methods does not use chemicals, it has high purity, but the equipment is complicated, uses a lot of energy, the concentration is not high and the quantity obtained is not large. Therefore, the cost is high and the field of use is limited.

2.1.2. The "bottom - up" approach

Physical methods can implement the principle of preparing AgNPs from the "bottom-up" by beams: gamma,³⁴⁻³⁷ electrons,³⁸ or microwave³⁹ activating components in solution to reduce Ag^+ of AgNO_3 salts into AgNPs.

According to the author group Bui Duy Du⁴⁰, the energy of gamma rays can affect the components of the medium such as water to form strong reactive agents including strong reducing agents such as H- radical with potential value - 2, 3 V:



Although the obtained AgNPs have the best shape and small size, the fabrication process must use different stabilizers^{34-37,40,41} and the maximum value of the UV-Vis spectrum ranges from 405.5 to 41.8 nm. With the advantage of using available equipment, the process of technology is not complicated and can prepare a large amount of AgNPs solution, so the cost will be more reasonable, but the resulting solution still has a large amount of NO_3^- ions, as well as other stabilizers and by-products, the field of application is only suitable for environmental remediation.

2.2. Chemical methods

2.2.1. Reducing agents

The chemical method of preparing AgNPs solution is to follow the principle from the "bottom-up" to create nanoparticles from the Ag^+ ions of silver salts by reducing the

reduction process.⁴² The commonly used silver salts are AgNO₃ and the reducing agents that have been used very different such as glucose (C₆H₁₂O₆),^{43,44} saccharose (C₁₂H₂₂O₁₁),⁴⁵ hydrazine (N₂H₄),⁴⁶⁻⁴⁸ ethylene glycol (C₂H₆O₂), ethanol (C₂H₅OH), aniline (C₆H₅NH₂),⁴⁹ sodium citrat (Na₃C₆H₅O₇),^{46,50-53} hydrogen (H₂),⁵² sodium borhydir (NaBH₄)⁵³⁻⁵⁷ ...

Table 1. Reducing and reactions to create AgNPs.

Reducing agent	Reaction equation	Size, nm	Ref
C ₆ H ₁₂ O ₆	C ₆ H ₁₂ O ₆ +2Ag ⁺ +2OH ⁻ →2Ag ⁰ +C ₆ H ₁₂ O ₇ +H ₂ O (4)	20.80 sphere	44
N ₂ H ₄	4AgNO ₃ +N ₂ H ₄ +4NaOH→4Ag ⁰ +N ₂ +4NaNO ₃ +4H ₂ O (5)		48
N ₂ H ₄	4AgNO ₃ +N ₂ H ₄ →4Ag ⁰ +N ₂ +4HNO ₃ (6)	8-50 sphere	46
RCHO	2AgNO ₃ +RCHO+2NaOH→2Ag ⁰ +RCOOH+2NaNO ₃ +H ₂ O (7)	10-250	49
C ₆ H ₅ NH ₂	C ₆ H ₅ NH ₂ +AgNO ₃ →Ag ⁰ +C ₆ H ₅ NH ₂ NO ₃ (8)	10-30	49
C ₆ H ₅ O ₇ Na ₃	4AgNO ₃ +C ₆ H ₅ O ₇ Na ₃ +2H ₂ O→4Ag ⁰ +C ₆ H ₈ O ₇ +3NaNO ₃ +HNO ₃ +O ₂ (9)		50
NaBH ₄	AgNO ₃ +NaBH ₄ →Ag ⁰ +1/2H ₂ +1/2B ₂ H ₆ +NaNO ₃ (10)	10-80	54-57
NaBH ₄	AgNO ₃ +NaBH ₄ +3H ₂ O→Ag ⁰ +7/2H ₂ +B(OH) ₃ +NaNO ₃ (11)	30-40	58

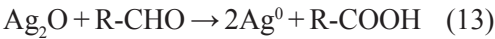
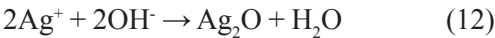
Table 1 presents the reaction equation to form AgNPs with a number of different reducers. To ensure the reduction process is completely done, the reducing agent usually has many times compared to silver salt. From the reactions in Table 1 it can be seen that in addition to the spherical silver nanoparticles after the reaction, there are ions of silver salt such as NO₃⁻, Na⁺, the products of reducing agents and stabilizers are added. Removing these Na⁺ and NO₃⁻ ions to obtain pure AgNPs is very difficult and expensive and also changes the properties of AgNPs.

Therefore, products containing ions are only applied in areas that do not require high purity of AgNPs.

Table 1 also shows that nanoparticles are obtained as a wide area, so it is necessary to use stabilizers to control the size of nanoparticles as desired. From Table 1, the reducing reaction mechanism according to different authors⁵⁴⁻⁵⁸ is also different. It means that the substances in AgNPs solution after the reaction will also vary, for example, NaBH₄ reduction reaction (10) creating B₂H₆⁵⁴⁻⁵⁷ gas will escape from the solution and if the reaction (11) quantity H₂ gas from the solution is 3.5 times higher than (10).⁵⁸

2.2.2. Stabilizers

The process of creating a silver nano colloidal solution with reducing agents that always exists in the system with ions and reducing agents, so silver colloids can be formed according to the equation:



and simulated as shown in Figure 2.^{44,54}

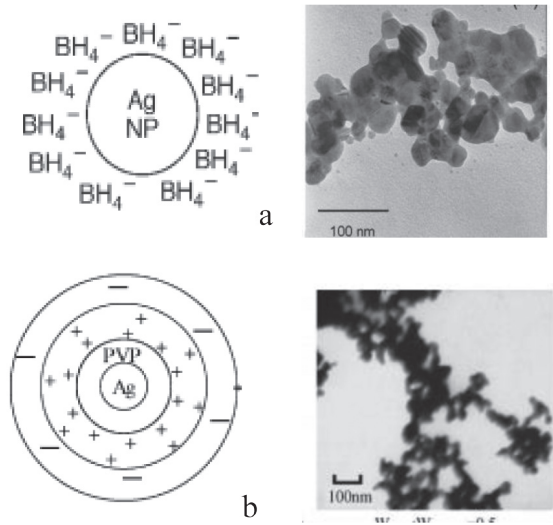


Figure 2. AgNPS colloidal seeds and stamp images made up of AgNO₃ chemical reduction with reducing agents: a) NaBH₄,⁴⁴ b) R-HO with PVP.⁵⁴ [Ref.].

In order to control the size and shape of AgNPs in the colloidal solution, it is not

formed into a large particles, stabilizers are high molecular compounds or surfactants added to the chemical reaction.⁵⁹⁻⁶¹ Stabilizers often have functional groups, dissolve well in the reaction environment, good compatibility or high biological activity, non-toxic and biodegradable ability.⁶² Table 2 is about presentation of some stabilizers often used for chemical manufacturing of AgNPs such as: chitosan,⁶²⁻⁶⁷ PVA,^{68,69} PVP,^{51,59,70} ...

Table 2. Stabilizers often used in the process of chemical manufacturing of AgNPs.

Stabilizers	Chemical formula	M _{ever} , g/mol	Ref.
Chitosan poly β(1,4)D-glucosamine cation		3,800-20,000	66
PVP polyvinyl-pyrrolidone		40,000	59
PVA poly vinyl alcohol		85.000	
PAA polyacrylic acid		15,000	
PAH poly allylamine hydrochloride		15,000	
CMC carboxymethyl cellulose		90,000	
NaDDBS Surfactnts (anion)		348	
SDS Surfactants (anion)		288	
TW80 Surfactants (neutral)		1,310	
CTAB Surfactans (cation)		365	

From Table 2, stabilizers can be found with electrical charge groups of straight or cyclic circuits, that can orient the adsorption on the AgNPs core to form a micell or reverse micell with the corresponding charge to combat flocculation of the colloidal system^{71,72} so that the stabilizers with the appropriate nature and concentration will control the size and shape of the AgNPs colloid as well as the characteristics of AgNPs as desired.

2.2.3. Silver nanocomposite

Fabrication of silver nanocomposite with chemical reducing processes will diversify AgNPs carried materials for applications in life. Composite materials carried AgPNs are usually studied as polymers PP, PET, Nylon, PC, ABS,^{73,74} PU,⁷⁵ PE,⁷⁶ ceramic, pottery,⁷⁷⁻⁷⁹ glass,⁸⁰ fabric, fiber,⁸¹⁻⁸³ paint.⁸⁴⁻⁸⁵ Common manufacturing methods are dispersed AgNPs made by chemical methods in materials, but can also be made *in-situ* from AgNO₃ with reducing agents in the material during the processing ceramics, fabric or polymers.⁸⁶⁻⁸⁸

2.3. Biological method

2.3.1. Microorganism

The biology method uses bacterial microorganisms, yeast, mushrooms, molds as AgNO₃ silver-deducted agents into metal silver and AgNPs^{89-91,119} microorganisms using silver salts as nutrients to survive and develop as described in Figure 3.

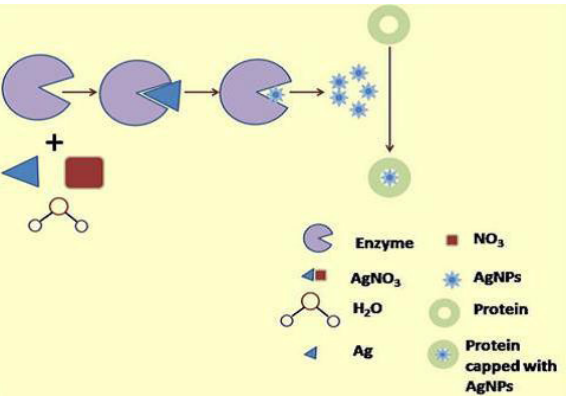


Figure 3. Microorganisms use Ag⁺ as a nutrient and reduce it to AgNPs.^{91,119}

From Figure 3 it can be seen that the protein can act as a stabilizer to control the size of AgNPs. There are many types of microorganisms studied and used to make AgNPs from AgNO₃ which are presented in Table 3.

The results from Table 3 show that microorganisms can reduce AgNO₃ salts to AgNPs with characteristic UV-Vis wavelengths from 380 to 460 nm and average particle sizes less than 100 nm. The special thing is that AgNPs products are stabilized with stable proteins for more than 6 months, so there is no need for stabilizers. However, the ions of AgNO₃ salt are still present in the reaction product, so the purity of AgNPs is not enough for application in the field of medicine.

Table 3. Particle size, characteristic UV-Vis spectra and references of some typical microorganisms using AgNPs preparation.

Microorganism	Size/UV, nm	Ref.
<i>Enterobacteria</i>	52.5 / 420-430	92
<i>Rhodopseudomonas palustris</i>	5-20 / 420-460	93
<i>Rhodobacter Sphaeroides</i>	9.56 / 420	94
<i>vibrio alginolyticus</i>	75 / 420	95, 96
<i>Halococcus salifodinae</i> BK6	50.3 / 380-440	97
<i>Bacillus</i>	42-94 / 450	98
<i>Euplotes focardii</i>	20-70 / 420	99
<i>Haloferax</i>	27.7 / 458	100
<i>Verticillium</i> (fungus)	25 / 420	101
<i>Aspergillus fumigatus</i>	5-25 / 420	102
<i>Penicilium</i>	5-25 / 430	103

2.3.2. Extraction solution – green chemistry

Humans develop in association with the plant environment and often use many types of plants for food or medicine, so using plants in the preparation of AgNPs is also a method with many advantages in terms of extremely rich raw materials, environmentally friendly and

low cost. Therefore, the method of preparing AgNPs by plant extracts has been studied all over the world such as USA,¹⁰⁴ China,¹⁰⁵ India,¹⁰⁶ Germany,¹⁰⁷ Africa¹⁰⁸ and Vietnam.¹⁰⁹ Water extracted from parts of plants such as leaves,¹¹⁰ roots,¹¹¹ bark,¹¹² tubers,¹¹³ flowers,¹¹⁴ fruits¹¹⁵ can all be used to prepare AgNPs. Table 4 presents extracts of some plants used to prepare AgNPs. Using plant water extract as AgNO₃ reducing agent to prepare AgNPs does not need to use more stabilizers, but the product is still available with NO₃⁻ ions and reducing products, so it also limits the application field.

Table 4. Extracts of some plants used to prepare AgNO₃.

The plants	Science name	Part	Ref.
Geraniums	<i>pelargonium graveolens</i>	Flower	104
Cordyceps	<i>Cordyceps militaris</i>	Total	105
Mud	<i>Brillantaisia patula</i> , <i>Crossopteryx febrifuga</i> and <i>Senna siamea</i>	Tree and leaves	108
Soybean	<i>soymida febrifuga</i>	Total	110
Carrot	<i>D. carota</i>	Tubers	111
Dill	<i>Syzygium cumini</i>	Total	112
Turmeric	<i>Curcuma Longa</i>	Tubers	113
Hibiscus	<i>Hibiscus Rosa</i>	Flower	114
Papaya	<i>Papaya</i>	Fruit	115
Basil	<i>Ocimum santum</i>	Total	117
Ginger	<i>Zingiber officinale</i>	Total	123
Tea	<i>Camellia sinensis</i>	Leaf	125
Sinus	<i>Azadirachta indica</i>	Leaf	124

Sesame oil (castor oil)	<i>Jatropha curcas</i>	Total	126
Euphorbiaceae	<i>Acalypha Indica</i>	Total	135
Mint	<i>Mentha piperita</i>	Total	122
Chrysanthemum	<i>Stevia rebaudiana</i>	Total	134
Amaranthaceae	<i>Chenopodium album</i>	Total	120
Fabaceae	<i>Casia fistula</i>	Total	133
Terminalia	<i>Terminallia chebula</i>	Leaf	118
C i n n a m o n , Camphor	<i>Cinnamomum camphora zeylanicum</i>	Bark	121
Garlic	<i>Allium sativum</i>	Total	127
Curry patta	<i>Murraya koenigii</i>	Leaf	132
Lemon basil	<i>Coleus amboinicus</i>	Total	131
Alfalfa	<i>Medicago sativa</i>	Total	130
Oranges, Lemons	<i>Citrus sinensis</i>	Total	128
Lemongrass	<i>Lemon grass</i>	Total	119
Bình bát	<i>Coccinia grandis</i>	Leaf	129
Combretaceae	<i>Terminalia catappa</i>	Leaf	128
Aloe vera	<i>Aloe vera</i>	Leaf	116
Lime tree	<i>Robustra</i>	Leaf	109

From Table 4, it can be seen that plants from all continents of the world are food sources and spices such as oranges, lemons, papayas, sesame, basil to pharmaceuticals such as cinnamon, garlic, lemongrass, and cordyceps as well as wood-bearing trees such as neem tree, etc., which can be extracted using water

containing AgNO₃ desalting agents into AgNPs. Common reducing agents in plant extracts are flavonoids, terpenoids, polyphenols, alkaloids, glucose which are compounds having carbonyl and hydroxyl groups or amine groups.¹³⁶

2.4. Electrochemical method

2.4.1. Role of electrolyte

The electrochemical method in the field of Physical chemistry can perform a top-down process by oxidizing the metal silver anode in the electrolyte into Ag⁺ ions with electrode potential value +0.799 V:¹³⁷



Simultaneously combined with the cathode reaction to reduce silver ions from the electrolyte to form silver nanoparticles, performing the bottom-up process:¹³⁸⁻¹⁴²



It is also possible to perform the preparation of AgNPs by simply reducing the reaction on the cathode (16) with AgNO₃ salt dissolved in the electrolyte solution and an inert anode such as Pt.^{143,144} To control the size of AgNPs obtained at the cathode, it is possible to use electrochemical parameters such as voltage, current density, conductivity as well as supporting measures such as pulses, ultrasound or will even produce strong gas release on the electrode at a higher voltage than conventional water electrolysis. With the usual electrochemical method, the electrolyte or anion NO₃⁻ of AgNO₃ still exists in AgNPs products, so it also limits the application field.

2.4.2. Role of applied voltage

Electrode reactions can occur in a non-electrolyte medium such as double distilled water with very low conductivity but the voltage must be sufficiently high^{138,139,141} or very high.¹⁴⁵⁻¹⁴⁹ There are two typical electrode arrangements in the electrochemical reactor when using high voltage (Figure 4).

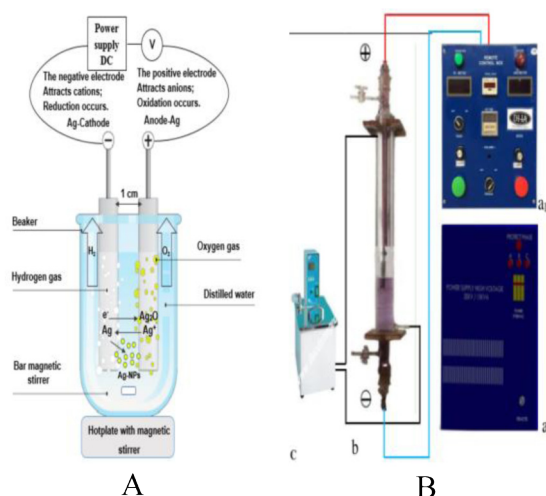
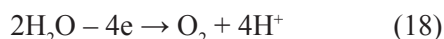
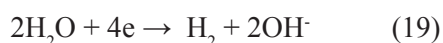


Figure 4. a) the cathode is parallel to the anode,¹⁴⁰ b) the bottom cathode is far from the upper anode¹⁴⁶.

With high DC voltage the potential drop across the electrodes will still be greater than the decomposition potential of water as well as the equilibrium electrode potential of Ag and the electrochemical oxidation on the anode to form Ag^+ ions as the reaction (15) as well as water is electrochemically decomposed to form O_2 :



At the same time on the cathode, the water will also be decomposed to form H_2 gas that escapes strongly towards the anode as shown in Figure 4b:



Due to the strong escaping gas covering the cathode surface, the amount of Ag^+ ions generated from the anode moves slowly due to poor conductivity, so it is difficult to reach the cathode to carry out the reaction (16). Therefore, the process of reducing Ag^+ to Ag^0 and then to AgNPs according to (17) will be carried out with new H_2 atoms generated from the cathode and dispersed into the solution:



Figure 5 shows the process of generating AgNPs by H_2 generated from the cathode by electrochemical reaction from high voltage. Figure 5a shows that the color of distilled water

is transparent, but after 3 minutes of reaction, H_2 gas escaping from the cathode turned white (b), and after 15 minutes of reaction, AgNPs formed turned dark color from the cathode side (c) and after 30 min the color of AgNPs occupied the entire reaction vessel (d).

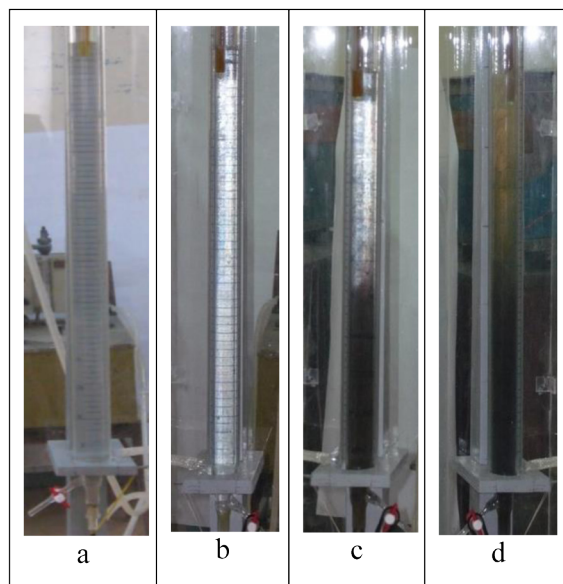


Figure 5. AgNPs generation process by high voltage electrochemical reaction.

With the method of electrochemical manufacturing AgNPs by high voltage DC in distilled water with Ag electrode, the obtained product still has a spherical shape, size smaller than 100 nm with UV-Vis spectrum at about 420 nm and the ability to kill all kinds of bacteria very good. However, the zeta potential is opposite in sign to the chemical method and has a high value, so there is practically no need to use a stabilizer. The conductivity of colloidal solutions is very small because there are no ions of the reactants, so the high purity is suitable for applications where only AgNPs are required.

2.5. Plasma method

Plasma is the fourth state of matter, the ionized state is changed from a gaseous state when further energized.¹⁵⁰ Unlike high-temperature plasma which produces a fully ionized state with only electrons and ions, low-temperature plasma ionization process only partially contains not only electrons, ions but also atoms, neutral

molecules and radicals and is being applied in many fields of science, technology and life.¹⁵¹ The cold plasma state is also used for the preparation of AgNPs by the reduction of AgNO₃ by free electrons or hydrogen atoms generated by plasma according to the reaction (16) or (20).¹⁵²⁻¹⁵⁴ Figure 6 shows the plasma generation methods for the preparation of AgNPs.^{152,153,155}

From Figure 6 it is shown that the gaseous medium can be used either by air on the surface (Figure 6A,b) or by blowing air between the two electrodes (Figure 6a and b) or by the ARC arc generating steam (Figure 6c).

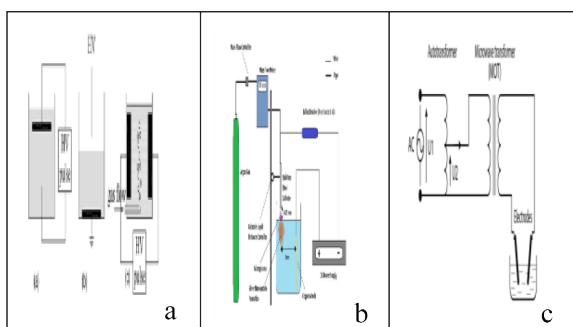
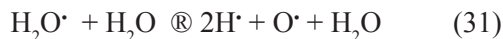
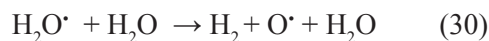
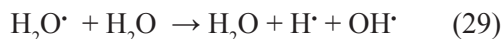
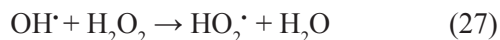
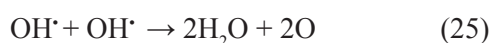
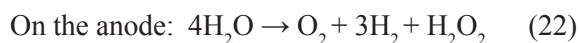
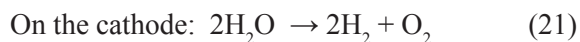


Figure 6. Principle of plasma generation for the preparation of AgNPs.

The plasma generation process uses electrodes and electrochemical reactions to create a gaseous environment when the electrodes are arranged as shown in Figures 4b and Figures 5 or Figure 6a, so the plasma method can also be considered as an electrochemical method with high voltage. In the plasma state, the water will be decomposed on the electrodes to create a large amount of gas that does not obey Faraday's electrochemical theorem as well as ionization reactions to create atoms, molecules and radicals:¹⁵²



UV rays in the presence of plasma also contribute to the radical reaction:



The reactants generated from the plasma medium can participate in the formation of AgNPs in addition to the reactions (16) and (20):

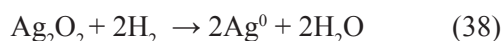
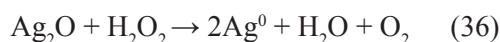
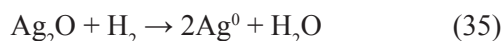
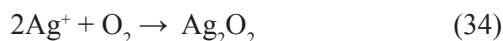


Figure 7 presents the process of generating AgNPs by high-voltage DC with the contribution of electrochemical plasma, showing that after the time of gas generation from the electrochemical reaction (Figure 7a), an anodic electrochemical plasma will appear after 15 minutes (Figure 7b) and the light yellow AgNPs color appearing from the anode towards the cathode gradually darkens over time of 23, 26, 35 min, respectively with Figures 7c, 7d and 7e.

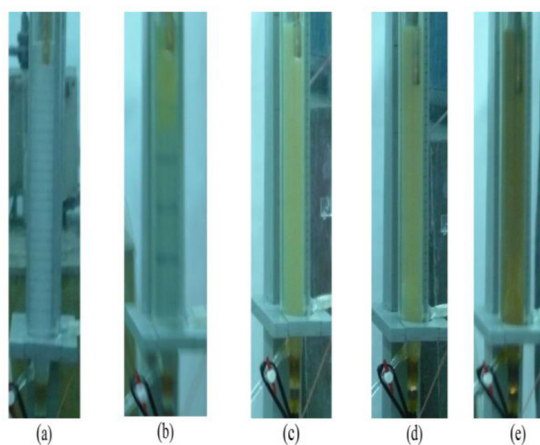


Figure 7. The process of generating AgNPs by high voltage DC with the contribution of electrochemical plasma.

The process of creating Ag_2O and Ag_2O_2 intermediates besides Ag^0 due to the presence of O_2 , OH^- , OH^\bullet , H_2O_2 agents, etc. in the electrochemical plasma environment has created a yellow color before turning black.¹⁵⁶ By Ronghen, EDX and XPS spectra also demonstrated the presence of O in AgNPs accounting for 5.77 , 9.6% and also increased the bactericidal efficiency of AgNPs.¹⁵⁷⁻¹⁶⁰ Similar to the method of preparing AgNPs by High voltage DC, electrochemical plasma contribution will create the ability to increase the speed, product concentration as well as the ability to kill bacteria, although there is a small amount of Ag_2O or Ag_2O_2 , but it does not affect the purity of the product.

3. CHARACTERISTICS OF NANOSILVER

3.1. Silver nanoparticles

The characteristics of shape and size of AgNPs were determined by imaging methods by electron microscopy SEM, TEM, FE-TEM. Particle size distribution was determined by statistical particle counting software from SEM or by Laser Scattering Particle Size Distribution Analyzer. Figure 8 presents TEM images of AgNPs shape and size (a) as well as particle size distribution from TEM (b) and laser determination (c). Figure 8 shows that AgNPs prepared by different methods all have near-spherical shape but different sizes in the nanometer region with Gaussian distribution as determined by laser method.^{154,158,161}

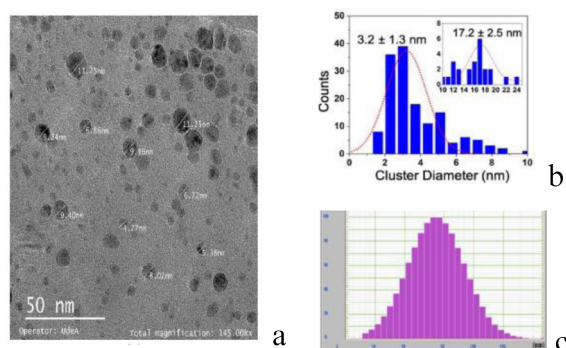


Figure 8. (a) TEM image of AgNPs, (b) particle size distribution counted from TEM image and (c) laser particle size distribution analysis.^{154,158,161}

X-Ray,¹⁶² XRD,¹⁵³ XPS^{142,152} methods are also used to further investigate the properties of AgNPs particles in terms of phase, ratio of elements or ions: $\text{Ag}/\text{Ag}^+/\text{O}$, contributing to a better understanding of state of AgNPs in solution.

3.2.1. Color

Figure 9 presents AgNPs products prepared by different methods such as: a) chemical,⁵⁴ or b) plasma.¹⁵⁵

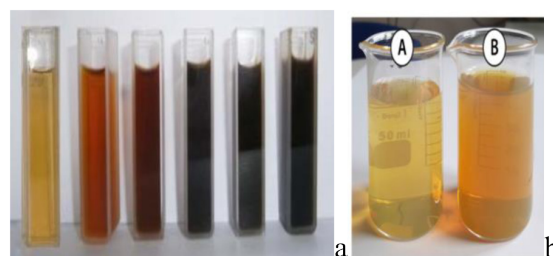


Figure 9. Color of AgNPs colloid a) chemically prepared,⁵⁴ b) by plasma at different times and concentrations.¹⁵⁵

The AgNPs products obtained are all true solutions in the state of a transparent colloidal system with color from light yellow to brown or black depending on the concentration and preparation time, while the colorless solution will have no AgNPs.¹⁶³

3.2.2. UV-Vis and zeta potential

The AgNPs colloidal solution has the important properties of UV-Vis plasmon spectrum and zeta potential. Figure 10 shows the UV-Vis spectrum,¹⁶⁴ zeta potential and colloidal particle size distribution.¹⁵⁵

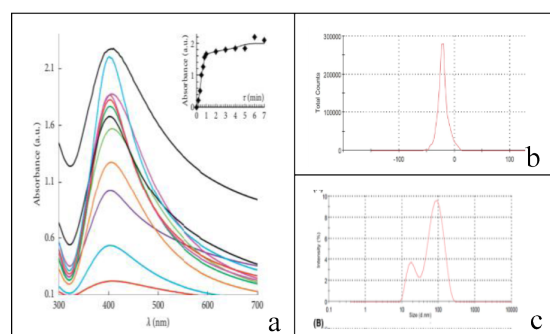


Figure 10. UV-Vis spectrum (a), zeta potential (b) and colloidal particle size distribution and (c) of AgNPs colloidal solution.

From Figure 10a, the UV-Vis spectrum can be found of the AgNPs colloidal solution that has a range of 400 nm and increases the height when the concentration or reaction time increases and the location is moved when the acacia grain nature is essentially affected. Figure 10b shows that the Zeta value is about -22.31 mV, proving that the surface of the AgNPs colloidal particles is positive and Figure 10c shows that the colloidal particles size is distributed from 20 to 90 nm. Therefore, Zeta is the diffusion layer, the surface of the colloidal particles should be dependent on the environment and charge of the AgNPs particles surface with values that change from yin and yang, but the absolute value is greater than 20 mV, the colloidal system will be durable over time. Table 5 presents the zeta value of AgNPs colloidal solutions with different stabilizers.^{60,66,139,159}

Table 5. The zeta value of some stabilizers.

Stabilizer	Chemical formula	mW, g/mol	ζ, mV
NaDDBS	C ₁₈ H ₂₉ SO ₃ Na	348	5, -30
SDS	C ₁₂ H ₂₅ SO ₄ Na	288	-2, -20
TW80	C ₆₄ H ₁₂ 4O ₂₆	1310	4, -15
CTAB	C ₁₉ H ₄₂ BrN	365	20, -30
PVP	(C ₆ H ₉ NO) _n	40000	0, -25
PAA	(C ₃ H ₃ NaO ₂) _n	14000	5, - 25
PAH	(C ₃ H ₈ ClN) _n	15000	5, 20
CMC	(C ₂₈ H ₃₀ Na ₈ O ₂₇)	90000	0, -10
Chitosan	(C ₆ H ₁₁ NO ₄) _n	20000	50 , 70
PP SH ¹⁶⁵	<i>Entada spiralis</i>	Chiết	-80,7
PPĐH ¹³⁹	DC 25 kV	Ag	-(27,39)
PPPL ¹⁵⁹	ARC discharge	Ag	-(40,70)

Table 5 shows that the value of zeta is very dependent on the nature of stabilization of chemical structure, weight, electronegativity,^{66,67} as well as depending on the modulation method and composition of ions that exist in glue solution.

3.2.3. Conductivity and pH

Because Ag⁰ or AgNPs silver particles are dispersed in water environments, it is impossible to conduct electronic conduct as metal as well as ionic forms like electrolyte solution. However, while using AgNO₃ in the methods as well as reducing the ionic amount of: NO₃⁻ as well as the reducing agent: Na⁺ or the products of the reducing agent will create the conductivity of the AgNPs solution. Moreover, AgNPs colloidal seeds adsorb ion and create charge.

Table 6. Electrical conductivity (χ, mS/cm) and pH of AgNPs solution are prepared by chemical and electrochemical methods.

Chem	c, ppm	RO	100	200	300	500
	χ, mS/cm	0,01	0,27	0,36	0,46	0,58
	pH	6,9	4,6	4,9	4,4	4,5
Electr	c, ppm	NC	109	185	285	411
	χ, mS/cm	0,003	0,08	0,07	0,07	0,07
	pH	6,9	6,64	5,63	5,78	5,79

Table 6 also shows that with the methods using of AgNO₃, conductivity levels increases when the concentration increases, but the methods of electrochemical or plasma use the silver electrode, the conductivity is small and has almost no change, even when the synthesis time as well as when the concentration increases.¹⁶⁶

3.2.4. Concentration of AgNPS

Determining the concentration of AgNPs is not as simple as determining the concentration of soluble substances because it is difficult to separate between silver nano and ionic. With AgNPs synthesis methods by using AgNO₃ often think that the process of reaction completely and the AgNPs concentration is also considered as AgNO₃ concentration. The AAS method transfers AgNPs to Ag⁺ so it cannot be determined by the nano form. With the methods of using Ag metal, it is possible to determine by soluble silver weight (c_{dm}) with the assumption that silver is

soluble for formation of AgNPs.¹⁶⁷ It can also determine the amount of AgNPs by adjectives. The amount of electricity according to the law of Faraday (c_{Far}), but besides the dissolving process, there are other electrochemical processes, so the concentration of Faraday's law is usually larger than the amount of soluble metal ($c_{\text{Far}} > c_{\text{Dm}}$).¹³⁹ The AAS method can also be used to determine the AgNPs concentration of the electrochemical and plasma modulation methods, but it cannot be separated from Ag^+ . The UV-Vis spectroscopic method for the determination of AgNPs alone would be the most accurate, but standard curve construction is not feasible because standard solutions are difficult to obtain.

3.3. Antibacterial ability

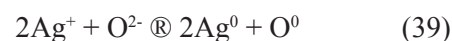
3.3.1. Traditional

Since BC, silver's bactericidal properties have been used for prevention and treatment of diseases such as: acupuncture needles, containers for liquids and drinking water for the prevention and treatment of infections. Former feudal dynasties in many countries around the world used spoons, knives, bowls and plates in eating and drinking to kill pathogens to ensure life safety. Silver has also been used for a long time in dentistry, to treat neurological diseases, eye diseases, to treat wounds, and to disinfect drinking water systems. During the World Wars, colloidal silver was used to fight gastrointestinal diseases and infections. From the late 19th century to the present, colloidal silver has been used quite widely in the form of oral and injectable drugs to treat arthritis, bronchitis, respiratory, lung, influenza as well as gastrointestinal diseases, stomach ulcers or Disinfection of purulent-necrotic burn wounds, dermatosis, boils or even syphilis, mastitis, meningoencephalitis, vestibular,...¹⁶⁸

3.3.2. Outstanding antibacterial properties

Elemental silver has outstanding bactericidal ability because Ag^+ ions exist in the form of salt.¹⁶³ In the form of AgNPs, with extremely large contact area, it is easy to provide Ag^+ , so the bactericidal efficiency is improved many times. Although there is still no consensus, the

bactericidal mechanism of Ag^+ aligned with 3 possibilities: (1) Destruction of the function of the cell wall; (2) Destruction of respiratory function due to inactivation of -SH group in O_2 transporter; (3) Destruction of DNA function by dimerization of pyridine interferes with DNA replication of bacterial cells. In addition, atomic oxygen is produced from the reaction:



It also inhibits the growth of bacteria. Furthermore, the plasmons of AgNPs are susceptible to thermogenesis and destruction of bacteria. Because of many different ways to kill bacteria, the bactericidal ability of AgNPs cannot be greasy or resistant like current antibiotics.

3.3.3. Antibacterials

Unlike antibiotics that are only suitable for bacteria, AgNPs can kill up to 650 types of bacteria, gram negative and positive as well as viruses and fungi, mold.¹⁶⁸⁻¹⁷⁰ Recent studies have shown AgNPs have remarkable anti-inflammatory and antiviral potential, even against viruses such as HIV^{171,172} or Sars corona,^{173,174} Monkeypox,¹⁷⁵ Hepatitis B,¹⁷⁶ Syncytial,¹⁷⁷ Herpes,¹⁷⁸ Tacaribe,¹⁷⁹ West Nile, Hanta, Nipah, Hendra, Chikugunya, as well as viruses of avian origin and pig.^{173,180}

3.3.4. Toxicity

The toxicity of silver and silver ions has been of concern for a long time due to the phenomenon of blue skin when the amount of silver accumulates and has not been eliminated in time.¹⁸⁰ With its small size, it is dispersed in gaseous and liquid environments and solids when used, AgNPs also easily penetrate into the body and accumulate in cells through the respiratory tract,¹⁸¹ esophagus or skin contact.¹⁸² Therefore, the toxicity of AgNPs is also very noticeable.^{183,184} Although AgNPs are not as toxic as ions, AgNPs still generate ions¹⁸⁵ from AgNPs and accumulate in organs such as lungs,¹⁸⁶ liver, and spleen¹⁸⁷ and cause harmful effects depending on the time and concentration of exposure¹⁸⁸ as well as the size and the shape of AgNPs.¹⁸⁹

4. APPLICATIONS

4.1. In chemistry

Silver metal as well as silver nano are applied due to its properties such as absorption and optical control, bactericidal, electrical and thermal conductivity, and especially as a catalyst for some reactions as well as as a sensor in analysis in chemistry.¹⁹⁰

4.1.1. Catalysis

The reduction of oxygen of epoxides to alkenes catalyzed by AgNPs can be 99% as efficient as using Au or AuNPs.¹⁹¹ AgNPs are used as catalysts for the reduction reactions of nitro aromatic compound,¹⁹² carbonyl as well as oxidation of alcohols, silanes, olefins, alkylation of amines and arenes as well as ring-opening or closing reactions and a variety of value reactions.¹⁹³ AgNPs are used as homogeneous or heterogeneous catalysts to synthesize many special chemical compounds with high efficiency such as:¹⁹⁴ pyrimido 96%, triazole 98%, pyrano 96%, isoxazole 93%, quinoline 88%, tetrazole 93%, benzopyranopyrimidine 95%, bivalent amine 92% , etc...

4.1.2. Analysis

With advantages in size, shape and surface, silver nanomaterials also play an important role in determining and controlling electrical, optical, physical and especially chemical properties. With the GC electrode combined with AgNPs, it is possible to have excellent electrocatalytic activation as a sensor for determining H_2O_2 in water with a concentration of 0.92 μM .¹⁹⁵ With different techniques, it is possible to fabricate the mounted electrode. AgNPs for performing cyclic voltammetry CV, differential voltammetry DPV, linear sweep voltammetry LSV, square wave voltammetry SWV analyzes with up to the limit of ppb detection of various organic compounds.¹⁹⁶ Especially, it has advantages in detecting chemical contamination in the state of the environment, so the number of publications by 2022 has been increasing rapidly.¹⁹⁷

4.2. In environmental treatment

The excellent bactericidal ability of AgNPs has been applied to environmental treatment mainly in three directions as surface disinfection, water disinfection and air sterilization.¹⁹⁸

4.2.1. Contact surface

Contact with material surfaces is the most frequent activity, so the antibacterial properties of AgNPs are also studied for applications in construction materials, fabrics or plastic tools. Interior paints with additive AgNPs 0.1 , 0.5 ppm have good antibacterial effect.¹⁹⁹ Glass surface coated with AgNPs not only has bactericidal value but also has plasmon effect to increase absorption capacity. energy.²⁰⁰ Plastic coated with AgNPs has many useful applications in medical transmission materials, in food packaging and preservation,²⁰¹⁻²⁰³ as well as export tropical fruits.²⁰⁴⁻²⁰⁶ Fabric fibers surface coated with AgNPs with the amount of 180 mg/kg have a bactericidal effect of 99.28%, even after 30 washing cycles it is still 98.77%.²⁰⁷

4.2.2. Water treatment

Water is necessary for the life of all things. Humans use water for all living activities as well as production, so they need clean water, but it is easy to pollute water sources with different wastes.²⁰⁸ AgNPs with special chemical and biological properties should be noticed. It is intended for use in environmental treatment systems including water.²⁰⁹ The European Union alone uses up to 20.5 tons of AgNPs to treat wastewater each year.²¹⁰ Effects of AgNPs in water treatment not only in the ability to kill bacteria but also in the chemical reaction ability²¹¹ as well as sensor application to control water pollution.²¹² In aquaculture, seafood AgNPs have also been used in water treatment to reduce pollution, infection as well as prevention of network diseases so as to have high economic efficiency.²¹³⁻²¹⁵

4.2.3. Air handling

The excellent bactericidal effect of AgNPs has also been studied for application to air

purification. By depositing AgNPs into a porous quartz tube fitted with an air purifier with a capacity of 250 m³/h, it is possible to both process organic compounds up to 91.6% butanol, 80% acetone, and 70.1% diethyl ether and 43% benzene as well as 99% bacteria and fungi and installed for E Hanoi hospital.²¹⁶ The air conditioning system combined with AgNPs to improved heat transfer ability saved energy on average 36- 58%.²¹⁷ However, when using AgNPs to treat air pollution, great care must be taken to limit the dispersion of AgNPs into the air so as not to cause inflammation of the respiratory system.²¹⁸ Therefore, the concentration of AgNPs to spray in the air should also be kept to a low level and avoid long exposure times.²¹⁹

4.3. In nanomedicine

AgNPs are widely used in many biomedical applications, known as nanomedicine including diagnostics, therapeutics, drug manufacturing, medical device coating, and personal healthcare. With increasing applications in medicine, a better understanding of the mechanisms is becoming necessary.²²⁰

4.3.1. Disinfectant

Because the hospital environment needs to be clean, the special antibacterial ability of AgNPs is noticed as a disinfectant agent for the environment as well as tools. The MBC concentration of AgNPs for hospital bacterial strains such as *S. Aureus* or *P. Aeruginosa* in the operating room after 20 minutes is 100 µg/mL and after 24 hours it is 12.5 µg/mL.²²¹ Fluid pathways or medical instruments are also tested for emergency disinfection with AgNPs.²⁰¹ Even the air in hospital rooms can be treated with contamination by bacteria as well as organic substances with AgNPs.²¹⁵

4.3.2. Diagnose

Silver nanoparticles are used in imaging diagnosis and treatment of dental and oral cancers, acting as a carrier to disperse to targets along with chemotherapy agents and as radiation and phototherapy enhancers. It is valuable

for studying inflammation, tumors, immune responses, and the effects of stem cell therapy, in which contrast agents are conjugated or surface-modified and bioconjugated to particles. nano. Silver has an important role in imaging systems with plasmonic properties that should produce a clearer image.^{222,223} Due to the reaction of AgPs with oxygen (ROS) of cancer cells, AgNPs have the effect of controlling and destroying DNA, contributing to the formation of nano cancer diagnosis and treatment in nano medicine.^{224,225}

4.3.3. Healing

The advantage of AgNPs is that they can kill many types of bacteria¹⁷¹⁻¹⁸⁰ and are not resistant to drugs like antibiotics,²²⁶ so special attention is paid to exploiting them to treat diseases. Disinfecting all types of open wounds²²⁷ especially in the treatment of burns²²⁸ or teeth and mouth²²⁹ with AgNPs not only heals the wound quickly but also leaves almost no scars after healing. With infectious diseases such as HIV, hepatitis, SARC, and chickenpox, injections with a concentration of 20 ppm of 10 nm AgNPs have achieved good curative effects.²³⁰ Because cancer is currently an incurable disease, AgNPs have also been researched and applied and found that cancer cells have been inhibited by AgNPs from proliferating as well as angiogenesis due to the destruction of living and proliferation conditions.²³¹ Furthermore, AgNPS particles have the ability to absorb heat, so they can use energy from the laser source to kill cancer cells.²³²

5. CONCLUSION

Silver nano is prepared by chemical, physical, biological and physicochemical methods. Raw materials for the preparation process are AgNO₃ salt and reducing chemicals such as NaBH₄, citrate salt, plant water as well as reducing microorganisms, or activating rays that create reducing properties of the solution such as γ. It is also possible to use Ag to disperse by laser or dissolve the anode into ions and then reduce it to form AgNPs. The appropriate purity for different practical applications of AgNPs products

depends on the method and materials used. Pure AgNPs solution is prepared by high-voltage electrochemical method or electrochemical plasma method because it only uses Ag and distilled water.

The basic characteristic of AgNPS is that the nanoparticle has a nearly spherical shape, the size is in the nanometer range and the UV-Vis spectrum is in the range of 420 nm with the height depending on the concentration and the pH value depending on the size. The zeta potential has an absolute value of ≥ 20 mV, which characterizes the stability of the silver nano colloid solution, then the negative or positive value depends on the method and the composition of ions in the solution. Pure AgNPs colloidal solution has a very small electrical conductivity, but the conductivity value will increase depending on the concentration of reducing agent ions or reaction products in the solution. A very important characteristic of AgNPs is the ability to kill microorganisms from positive and negative bacteria, viruses to fungus by destroying cell membranes, affecting -SH groups as well as destroying functions microbial DNA.

AgNPs are applied in chemical fields as catalysts and analytical sensors. In the environment, AgNPs are applied to treat bacterial infections as well as air and water pollution. In medicine, AgNPS is given special attention in research and application to treat environmental infections, medical tools and equipment; diagnose and heal many diseases, including dangerous diseases such as, burn, HIV, SARC and cancer.

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