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Preparation and Characterization of Silver Nanoparticles by Chemical Method for Modification with Polyvinyl Chloride Electro Spun Nanofibers

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Abstract

In this study, a chemical procedure was used to produce nano-silver particles (reduction method). At different molar concentrations, reactive substances including sodium citrate and silver nitrate were employed. A smaller average size is produced by mixing (sodium citrate: silver nitrate) at an optimal molar concentration of (1:20). To determine the Zeta Potential and (PDI) of the generated silver nanoparticles, the (DLS) Zetazaser instrument was employed. To examine the structural and component of produced silver nanoparticles, X-ray diffraction and EDX techniques are applied. The outcomes showed that this approach generated stable, homogenous nano silver particles with an average size of 58 nm and a PDI factor of 0.055. The electrospinning process was used to create different nano-fiber diameters of poly vinyl chloride (PVC) nanofibers reinforced with silver nanoparticles from solution under strict working conditions, including solution parameters (viscosity, temperature). Gram-positive and gram-negative microorganisms were used to test the antibacterial activity of tissue generated with silver nanoparticles. According to the findings, both species of bacteria were effectively suppressed to a degree of 99.99 percent by the tissue that was created.

1. Introduction

The topics covered by nanoscience and nanotechnology are numerous. It is focused on things with nanometer-scale dimensions. Active nanoparticles and polymers can be used to improve performance and open up new opportunities for the use of lightweight materials [1]. Due to their unique electrical, optical, mechanical, magnetic, and chemical properties compared to bulk materials, noble metal nanoparticles have attracted a lot of attention in recent years [2]. They have unique and unusual characteristics because of their small size and large specific surface area. Due to their characteristics, metallic nanoparticles have found use in a number of fields, including catalysis, electronics, filtration, purification, food packaging, medicine, and photonics [3-7]. The idea of using silver nanoparticles as an antibacterial agent is quite recent. Due to their high reactivity and enormous surface to volume ratio, nanoparticles are efficient at preventing the development of bacteria in both aqueous and solid mediums.

Silver-containing substances can be used to purify water or destroy bacteria on fabrics [8]. For the creation of metallic nanoparticles, many techniques including chemical vapor deposition, micro emulsion, electrochemistry, thermal breakdown, laser ablation, and microwave irradiation [9] and others have been documented. The simplest and most popular bulk-solution synthesis method for metal nanoparticles was chemical reduction of metal salts. In fact, several shapes and sizes of nano-sized metal silver particles have been discovered through the chemical reduction of silver salts. [10]. This synthetic process uses a variety of reducing agents to reduce an ionic salt in a suitable medium while a surfactant is present [11]. Electrospinning is the most efficient method for fabricating nanofibers and nanocomposite textiles, which are common in nanotechnology due to the production of nanofibers with diameters ranging from 2nm to 5m. A syringe pump, a high voltage root, and a roller or continuous collector are the essential components of this technique. The produced polymer solution is injected and boosted in steady flow with a syringe pump, and the needle is connected to a high voltage root in the range of 3kV -30kV. Electrospinning is affected by a variety of factors, including solution parameters such as viscosity, surface tension, and molecular weight, instrumental parameters such as feed rate, needle-collector distance, and voltage source, and ambient parameters such as humidity, temperature, and weathering effect. Each of the above mentioned characteristics has a unique effect. [12-13]. On the other hand, the morphology and efficiency of nanofibers are heavily influenced by the characteristics of polymer solutions. Viscosity is critical in electrospinning, which is determined by adding polymer concentration to solutions. The molecular weight and concentration of the polymer have an impact on viscosity. Surface tension is another significant agent effect on electrospinning operations, which is influenced by the composition of polymer solutions and results in a change in surface tension. When the ratio of free solvent molecules is high, molecules mix together and form beads [13-19]. At the nanometer scale, nano composites are a combination of two phases of materials, the first of which is a matrix phase and the second of which is a reinforcing phase [20].

The creation of silver nanoparticles and their usage in bacterial suppression applications have been the subject of numerous prior investigations. Silver Nanoparticles (NPs) were created via chemical reduction in 2010 by Kheybari S. et al. for use in antimicrobial applications. (NPs) reduction of silver nitrate in the presence of poly [N-vinylpyrolidone] (PVP) as a stabilizer and a reducing agent. Scanning electron microscopy (SEM) and a laser particle analyzer both confirmed the nanostructure and size of the silver NPs (LPA). UV spectroscopy was employed to examine the silver nanoparticles' forms. By measuring the minimum inhibitory concentrations (MIC) of both Gram positive (Staphylococcus Aureus and Staphylococcus Epidermidis) and Gram negative (Escherichia Coli and Pseudomonas Aeruginosa) bacteria, researchers were able to determine the anti-bacterial activity of silver nanoparticles. The findings show that it is possible to make (NPs) with a diameter of 50 nm that are both very powerful anti-bacterials [21].

A review study on the use of silver nanoparticles and electrospun nanofibers as an antibacterial agent was published in 2021 by Luis Jess et al. Silver nanoparticles have a well-known antibacterial bioactivity, and they are broadly applicable in a variety of applications, playing a crucial role in the biomedical sector. The electrospun nanofibers, on the other hand, have qualities that can improve the application of silver nanoparticles. The loading of silver ions into electrospun nanofibers, however, affects how bioactive silver nanoparticles are. This review compares various silver incorporation techniques into electrospun nanofibers and their antibacterial efficacy, discusses the drawbacks of each technique, and highlights the most promising one. Due to their ease of use and effective outcomes, this review demonstrated that direct mixing and ultraviolet irradiation procedures were the preferable approaches for adding silver nanoparticles. Additionally, polyacrylonitrile nanofibers (PAN) have been the system having silver nanoparticles in it that has been published on the most. Finally, silver nanoparticle-loaded nanofibers have strong antibacterial activity regardless of the approach used [22].

Irena and Tomasz released an article in 2022 regarding the need of employing nanofibers with antibacterial activity because many pathogens have developed broad drug resistance. By adjusting variables like solution/melt viscosity, feeding rate, and electric field, electrospinning is a versatile technology for creating ultrathin fibers with desired qualities. Low viscosity and fast feeding rate produce fiber discontinuities or droplet formation, while high viscosity and moderate feeding rate block the spinneret. High field strength reduces the time required for the fluid streams to solidify, whereas low field strength prevents the Taylor cone from forming, hence the electric field must be set up properly. Temperature, humidity, and the environment all have an impact on electrospinning. Significant progress has been made in the creation of electrospun nanofibers for diverse applications as well as the

development of electrospinning techniques in recent years. This review discusses the most recent work on using electrospinning to create composite polymer fibers with antimicrobial properties by incorporating well-defined antimicrobial nanoparticles (such as silver, titanium dioxide, zinc dioxide, copper oxide, etc.), encasing traditional therapeutic agents (antibiotics), plant-based bioactive agents (such as crude extracts and essential oils), and pure compounds (such as antimicrobial peptides and photosensitizers) in polymer nanofibers with Co The studies show that electrospinning is a successful method for producing antimicrobial fibers for the biomedical, pharmaceutical, and food industries [23].

The aims of this objective is to manufacture silver nanoparticles using a simple chemical process in order to use them in antibacterial tissues for industrial, medicinal, and biological purposes.

2. Experimental Part

2.1. Material and Methods

Silver nanoparticles were prepared using silver nitrate (AgNO3), hydrazine hydrate, sodium citrate, and sodium dodecyl sulphate (SDS), all of which were acquired from Merck Peruana, as well as de-ionized water as a solvent media was used.

2.2. Preparation of Silver Nanoparticles via Chemical Reduction Method

Fig 1. Show the diagram of silver nano particles preparation steps. Two different weight ratios of reaction materials S.S + S.N were used to make three different sizes of silver nanoparticles: (1:9), and (1:20). Nano silver particles were perorated using deionized distil water (DDW). After heating AgNO3 and DDW to boiling, tri-sodium citrate was added drop by drop. The solution was forcefully stirred throughout the procedure. The solution was swirled until it reached room temperature after 2 hours of heating. The nano particles were subsequently separated from the water using a centrifuge (Hettich 1401-01 Universal 320 Classic Benchtop Centrifuge, 346 x 395 x 520mm (H x W x D), 1 to 99 min, 59 sec, 15000 rpm). The color preparation is shown in Figure 2. Nano silver particles.

2.3. Preparation of PVC Electro Spun Nano Fibers and Their Nanocomposites

For the manufacture of PVC Nano fibers, poly vinyl chloride (PVC): $(CH_2CHCl)_n$ was employed. It was purchased from Sigma-Aldrich chemistry in the United States as a white powder with an average molecular weight of Mw = 85,000 and a density of 1.4 g/mL at 25 ℃. 4- Tetra hydro furan (THF) was employed as a PVC solvent having a boiling point of 66 degrees Celsius. (Qualikems Fine Chem. Pvt. Ltd) Nandesari, Vadodara, India provided it. The composite nanofibers were made by adding the amounts listed in Tables (1) to a 13 percent polyvinyl chloride solution, according to the pumping conditions listed in Table (1).

Table (1). The conditions of electrospinning process.

PVC /THF con. w/v	Solution parameters			Processing parameters	Composites nanofibers AgNPs %
	Viscosity	Electrical conductivity µS/cm	Surface tension		
0.1	1.5	0.1	17.68	HVPS = 15 kV Distance =20 cm Flow rate = 0.5ml/hr Needle diameter = 0.4 mm	
0.13	2.9	0.1	18.07		0.2:0.998 0.7:0.993
0.15	4.5	0.1	18.97		

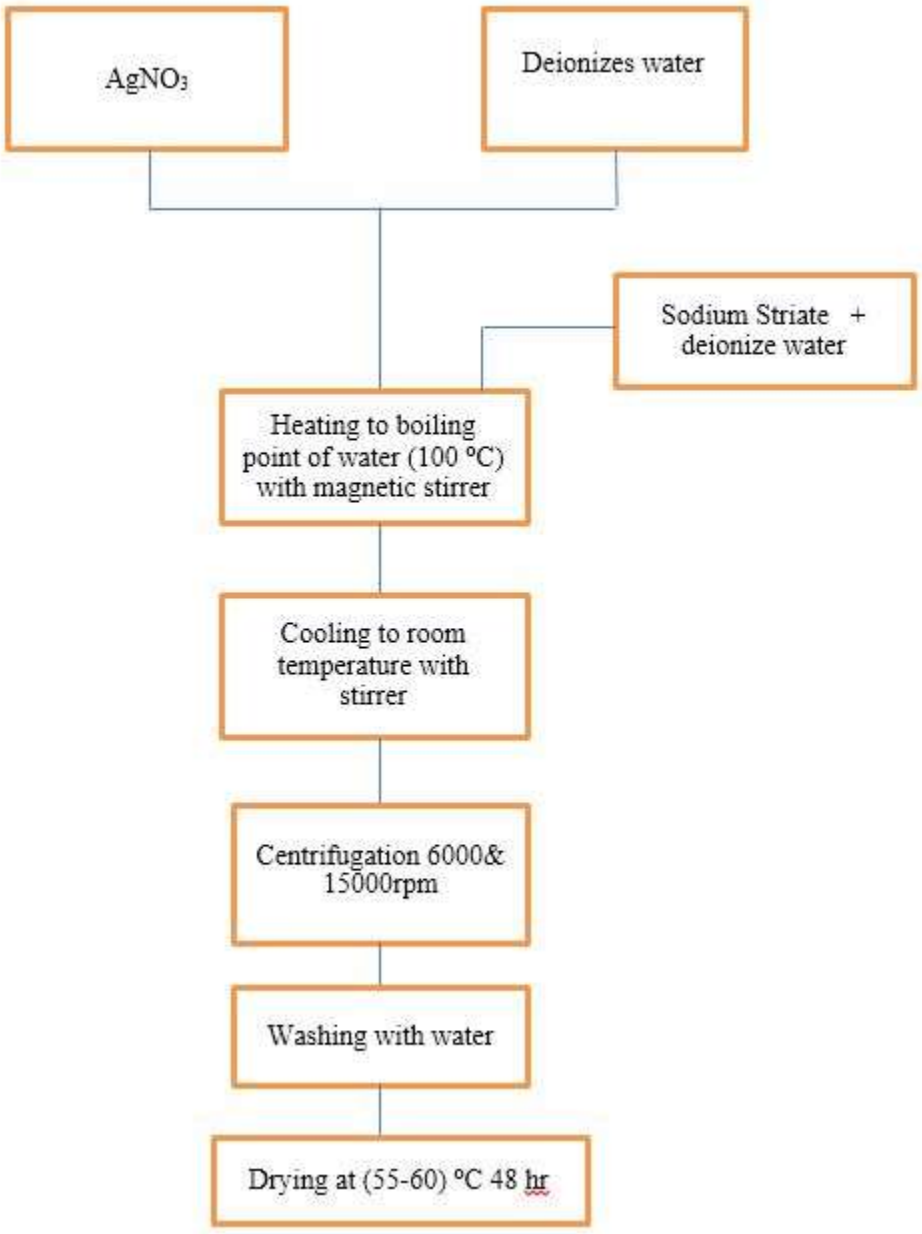
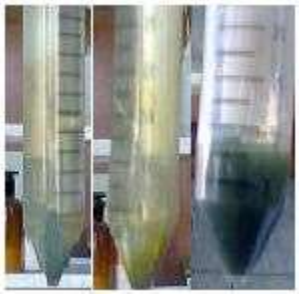


Figure (1). The steps of preparation of silver nanoparticles.



a **b** **c**

Figure (2). Silver nanoparticles preparation a. after 1 hr of reaction b. after 3 hr. of reaction c. drying Ag NPS.

2.4. Setup of Electrospinning Technique

Figure (3) depicts the NANOAZMA electrospinning device, which was utilized to prepare nanofibers.

Figure (3). NANOAZMA electrospinning system.

For controlling the flow of fluid through the needle, a syringe containing polymer solution was placed in a syringe pump. A positive HV electrode was connected to this needle. The HV's negative electrode was linked to the metallic collector, which was then connected to the earth. Table 1 lists the solution and processing parameters that were employed to make the electrospun nanofibers.

3. Characterization Technique

X- Ray diffraction and EDX techniques used to determine the purity of Ag nanoparticles , DLS, ZETAZIZER were used to determine the zeta potential, size average, and (PDI) of the resultant nanoparticles, SEM microscopy were used to determine the morphology of silver nanoparticles, and SEM was used to investigate the electrospun nanofibers. Antibacterial test was performed by two technique, the first method was performed by Agar well diffusion method to measure the inhibition diameter zone, and the second method was [Antibacterial Finishes on Textile Materials: Assessment of AATCC test method 100-2004] was used to test and assess the degree of antibacterial activity of textile nanofibers. The antibacterial activity of fabric substances was calculated using Equation (1) [21].

$$\eta = \frac{N_1 - N_2}{N_2} \quad \ldots (1)$$

Where N1: number of surviving bacterial colonies from the controlling sample, N2: number of surviving bacterial colonies from the test sample.

4. Results and Discussion

4.1. Zeta Potential and DLS.

The best reactive material mix ratio was (1:20) to (S.S: S.N), which resulted in very homogeneous Nano silver with an average size of 58 nm, except for one peak, which corresponds to a 62 nm average size, demonstrating the homogeneity of the produced nanoparticles. According to the (DLS) ZETASIZER findings, Figure (4a).

As well as the (DLS) results, which refer to obtain Nano silver particles with very small zeta potentials (-35.5), this refers to polydispersity of nanoparticles is very high, which refers to stability of nanoparticles for a long time without any reaction and bonded between them, this is also proven by (PDI), which is (0.055) as shown in Figure (4b), is very small and very close to zero value, this refers to high stability of (Ag) Nanoparticles [25].

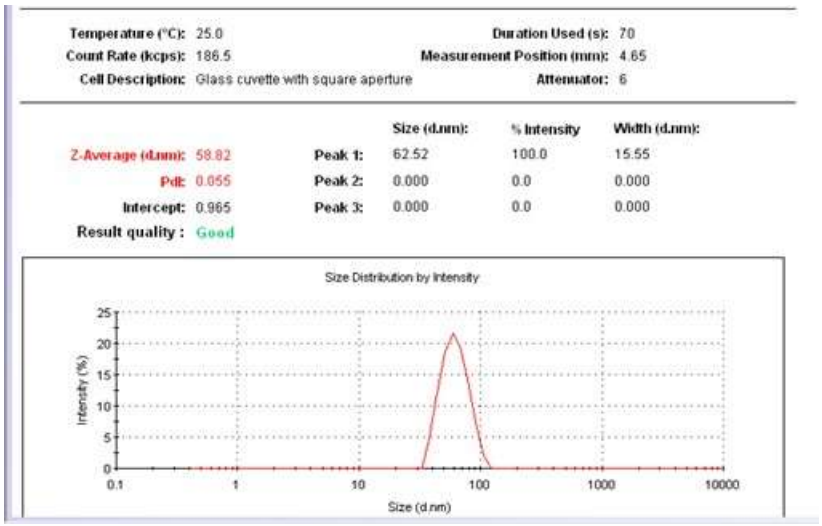


Figure (4a). Particle size of Ag NPs.

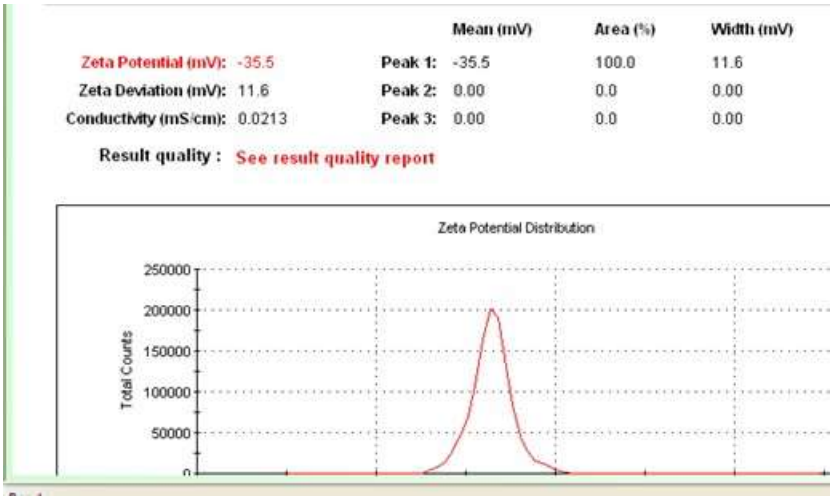


Figure (4b). Zeta potential of nano silver.

Figure (4a & b). (DLS) ZETASIZER Results of Nano Silver Particles

4.2. X-Ray Diffraction

The spectrum of the nano-silver prepared in this work and the standard X-ray diffraction of Ag nanoparticles are shown in Figures 5 (a) and (b), respectively.

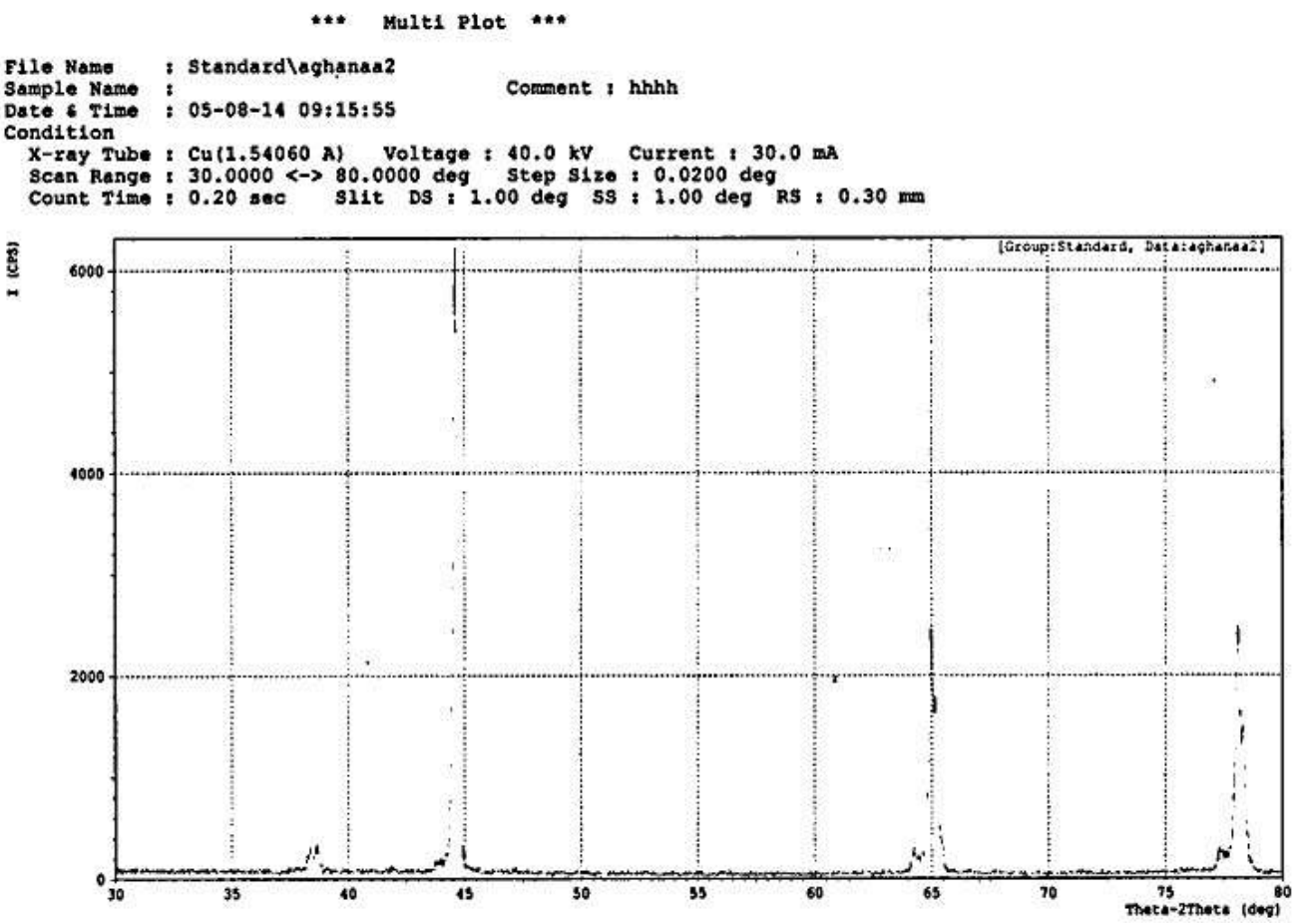


Figure (5a). XRD spectrum of prepared Ag nanoparticles.

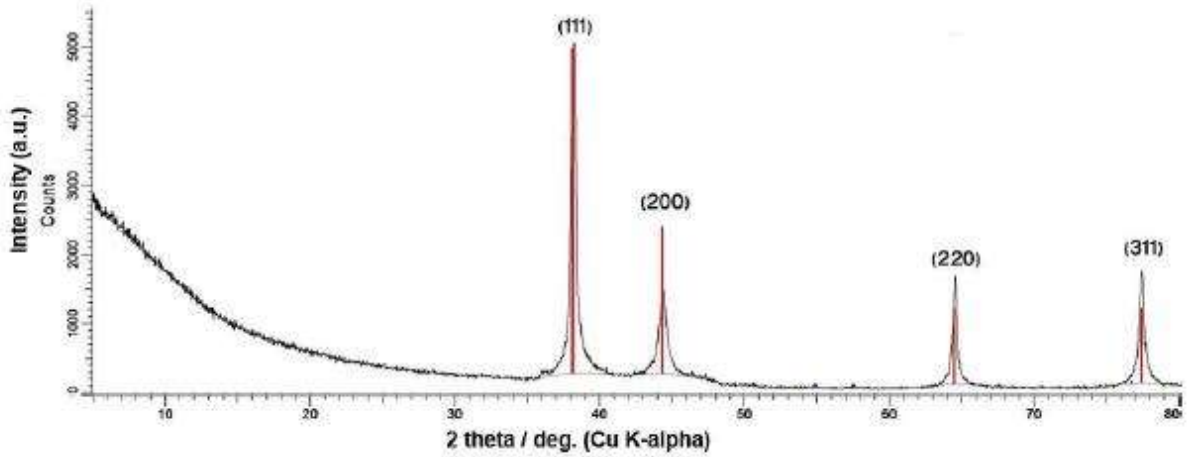


Figure (5b). Standard XRD spectrum of Ag nanoparticles [23].

X-ray diffraction (XRD), which has long been used to define and identify standard materials and nanoparticles, was employed to further characterize the structural characteristics of AgNPs with reference to phase composition and crystallographic orientation [26]. The produced Ag nanoparticles' X-ray diffraction shows excellent agreement with the standard curve (fig. 5b). The synthesized Ag nanoparticles' recognized peaks are 38, 44.3, 65, and 78. The standard curve's [38.23 (1 1 1), 44.41 (20 0), 64.38 (2 2 0), and 77.5 (3 1 1)] detected peaks, which showed the crystalline nature of the produced particles (Figure 5a & b). The produced Ag nanoparticles' X-ray diffraction shows excellent agreement with the standard curve (Figure 5b).

4.3. SEM Morphology of Silver Nanoparticles

Figure (6a) shows the SEM picture of resulting Nano silver. The SEM pictures of Nano silver generated under (1:20) of ($AgNO_3$: $C_6H_5O_7Na_3$) and a higher centrifuge speed of roughly 15000 rpm, Figure (6) shows the silver nanoparticles samples that prepared by. According to zetasizer data, the Nano silver average size is (58 nm), the PDI factor is (0.055), and the zeta potential is (-35.5), indicating that the sample is relatively stable and does not likely to form agglomerations. The SEM results of Ag nano particles show that Ag NP are homogeneous and that no silver granulate is present. Additionally, SEM explicitly confirmed the presence of very small Nano silver particles, which is due to its low PDI factor and high negative zeta potential, as shown in Figure (4).

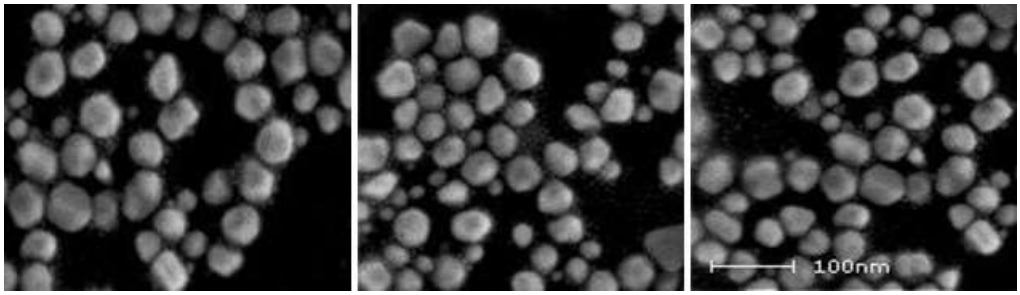


Figure (6a). SEM image of silver nanoparticles with mix ratio (1:20) of (S.S:S.N).

The amount of materials involved in the reaction, as well as the reaction conditions of time and temperature, have a significant impact on the morphology and nature of the materials produced by the chemical reduction method, as the persistence of the reactants in the reaction medium for an extended period of time increases the granular size of the resulting nanomaterials due to continued crystal growth of clusters, resulting in agglomerated nanomaterials. This is clearly demonstrated in Figure (6b), which shows SEM pictures of the second sample after a 6 hour reaction time.

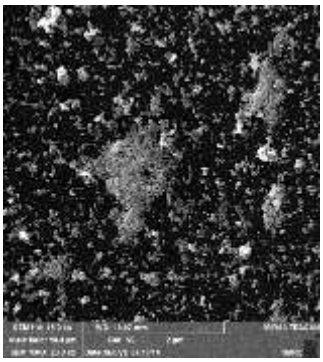


Figure (6b). SEM of silver nanoparticles after 6 hr. of reaction.

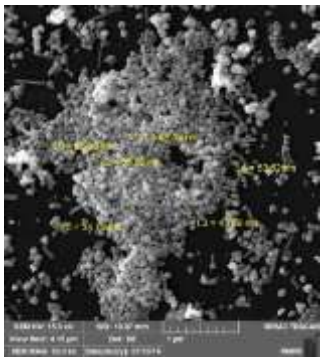


Figure (6c). SEM of silver nanoparticles by mix Ratio (1:9) of (S.S:S.N) and 3hr. reaction time.

Figure (6). The SEM Images of Resulting Nano silver.

Figure (6C) displays the SEM of the sample produced by the reactant mixing ratio (1:9). Because of its instability, the resulting particles clump together when the ratio of sodium citrate increases and the ratio of silver nitrate decreases [25-26].

4.4. EDX analysis

The generated silver nanoparticles are examined by EDX in Figure (7), which demonstrates that they are extremely pure and consist of 99 percent silver and just 1% oxygen particles as a result of the reaction process. This finding is consistent with Hongshui Wang et al. 2005 [27].

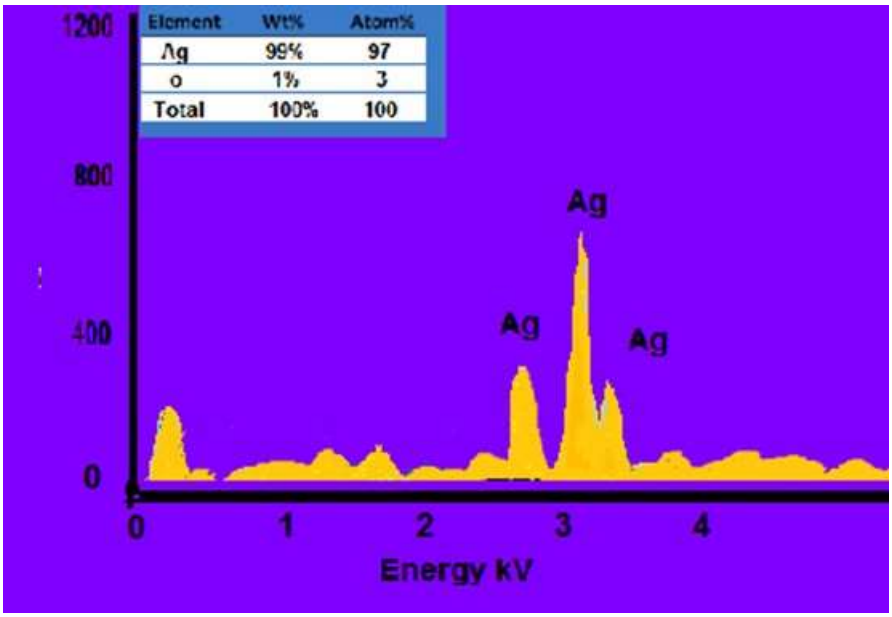


Figure (7). EDX of prepared nano silver.

4.5. Electro spun PVC Nanofibers

SEM images of fibers formed by increasing the concentration of polyvinyl chloride/tetrahydrofuran solution are shown in Figure 8a-c, and the diameter and morphology of the fibers are listed in Table (2).

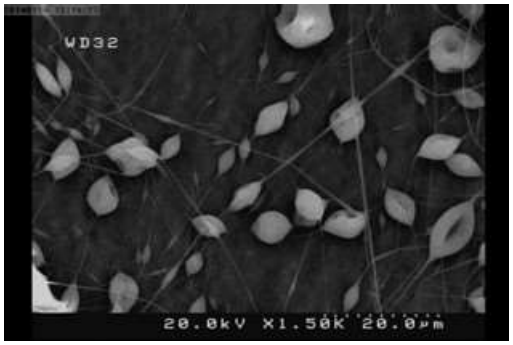


Figure (8a). SEM of (PVC) NF. Con. 0.1 w/v.

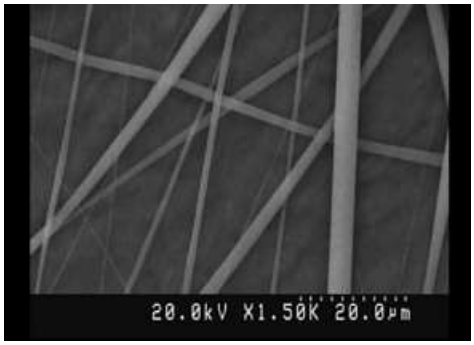


Figure (8b). SEM of (PVC) NF. Con. 0.13 w/v.

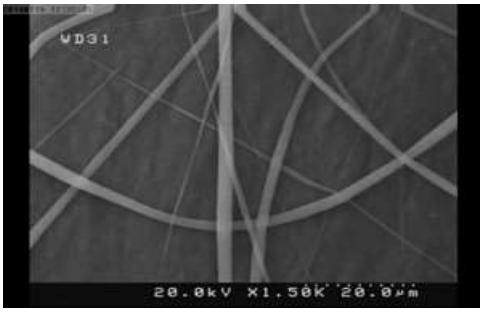


Figure (8c). SEM of (PVC) NF. Con. 0.15 w/v.

Table (2). Diameter and morphology of PVC electro spun nanofibers.

Con w/v	PVC M.F.D nm	Morphology
0.1	156	More elongate &prune beads
0.13	703	No beads
0.15	1094	no beads

Observe figure (8 (a-c)) and table (2). The product is a mixture of beads and nanofibers with a diameter of roughly 156 nm, as determined by the solution concentration (0.1 w/v). The existence of beads is thought to be related to axisymmetric instabilities in the jets flow [28]. When the concentration was increased to (13% con.), the result was smooth and homogeneous nanofibers with no beads and larger Nano-diameters (703 nm) [18]. Microfiber diameter grew to (1094 nm) with no beads when concentration was increased to 15% con. w/v. [29]. As demonstrated in table 3, raising the concentration causes the viscosity and surface tension of the solution to increase. Because a higher concentration of polymer chains in solution means a higher number of polymer chains in solution, this can lead to an increase in viscosity and surface tension, and vice versa, this can also lead to an increase in nanofiber diameter since keeping the other processing parameters constant. [13 & 30]

4.6. Antibacterial Activity

4.6.1. Antibacterial Finishes on Textile Materials and Degree of Antibacterial Activity

Antimicrobial activity was measured according to Escherichia coli by the Antibacterial Finishes on Textile Materials: Assessment of AATCC test method 100-2004. The antibacterial activity of PVC/AgNPs Nanocomposite fibers made by elctrospinning technique at this study against two types of bacteria is shown in Table (3) and Figures (9 a-d) and (10 a-d). S. aureus as a positive type and E. colie as a negative type.

Table (3). Show the antibacterial efficiency of PVC/AgNPs versus S. aureus and E. Colie.

PVC/AgNPs con. %	CFU/ML	E% versus *E. colie*	E% versus *S. Aureus*
0.2:0.998	*E. Colie* $1.3*10^3$ *S. Aureus* $2.3*10^3$	50%	40%
0.7:0.993	*E. Colie* $1.3*10^3$ *S. Aureus* $2.3*10^3$	99.99%	99.99%

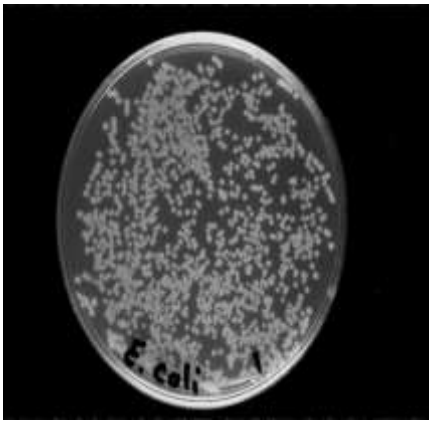


Figure (9a). Control sample.

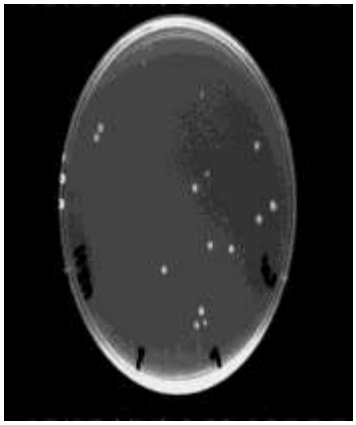


Figure (9b). Test sample.

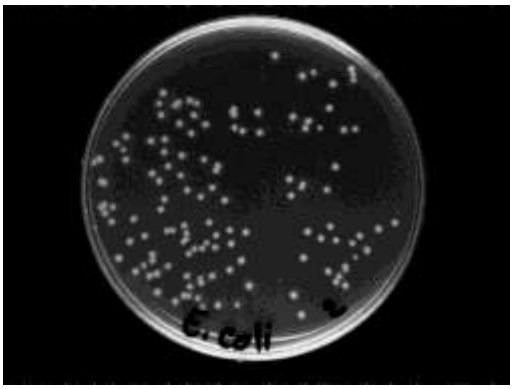


Figure (9c). Control sample.

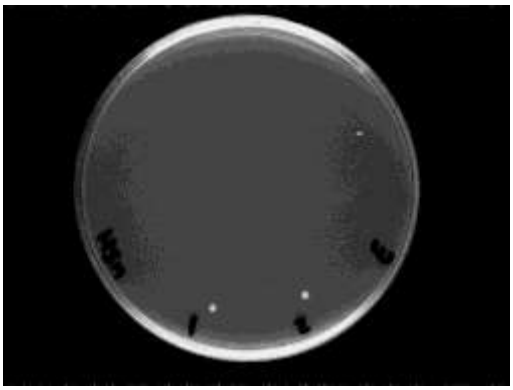


Figure (9d). Test sample.

Figure (9). Antibacterial activity of AgNPs/ PVC nanocomposite fibers versus E. colie with a, b 0.2%:0.998 ratio of AgNPs and c, d 0.7%:993.

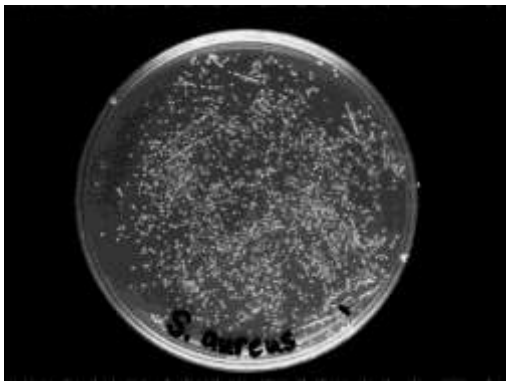


Figure (10a). Control sample.

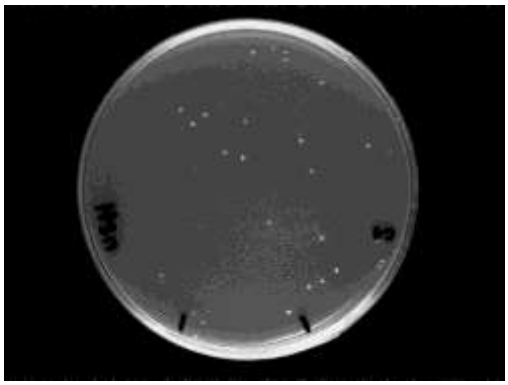


Figure (10b). Test sample.

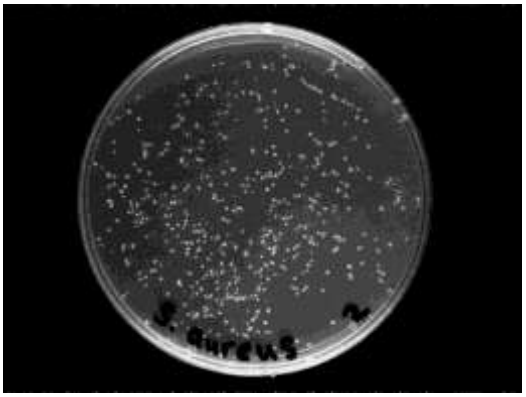


Figure (10c). Control sample.

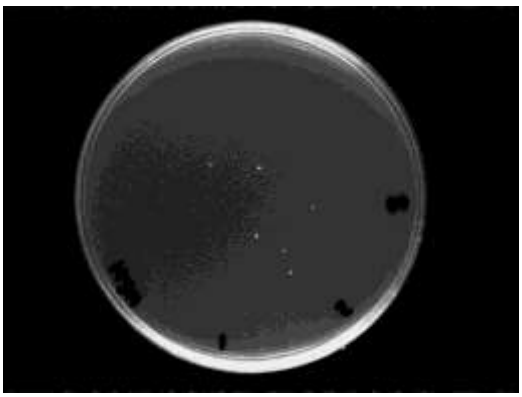


Figure (10d). Test sample.

Figure (10). Antibacterial activity of AgNPs/ PVC nanocomposite fibers versus S. Aureus with a, b 0.2%:0.998 ratio of AgNPs and c, d 0.7%:993.

The activity of PVC/AgNPS is very strong, as shown in previous figures, and it increases when the concentration of AgNPs in Microfibers increases. To survive, bacteria, viruses, and fungus all rely on an enzyme to consume oxygen. Silver inhibits the enzyme's function and prevents oxygen intake, killing the bacteria. [30]. We also note that its ability to suppress gram-negative bacteria is greater than that of gram-positive bacteria, but when the percentage of silver nanoparticles is increased to 0.7 percent, the inactivation effectiveness for both types of

bacteria reaches 99.99 percent. This is because gram positive bacteria have a wall thickness of 20-30 nm, whereas gram negative bacteria have a wall thickness of 10-12 nm.

4.6.2. Antibacterial Inhibition Zone

Mueller Hinton (MH) agar was prepared, poured into Petri dishes, and allowed to cool down before each microorganism was inoculated on the surface of a different MH agar plate to test its antibacterial activity against Staphylococcus aureus and Escherichia coli, representing gram-positive and -negative bacteria, respectively. The agar-well diffusion method was used to determine the antibacterial activity. Due to the nature of Ag nanoparticles as an antibacterial agent, Figure (11) and Table (4) showed how the antibacterial activity and diameter of inhabitation of samples increased following the addition of Ag nanoparticles.

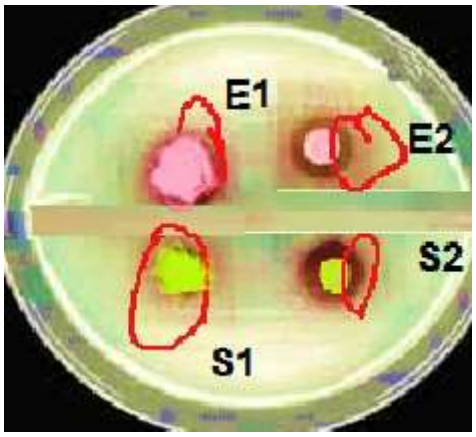


Figure (11). Inhibition zone of antibacterial test.

Table (4). Inhibition diameter of antibacterial test.

Sample No.	E. *coli* Zone Diameter (cm) E1,E2	S. *aureus* Zone Diameter (cm) S1,S2
1	0	0
2	1.3	1.2

We observe that silver nanoparticles have an antibacterial effect. This effect may be related to the production of reactive oxygen species, the release of Ag+ ions from AgNPs, which denaturize proteins through their interactions with sulfhydryl groups, and the attachment of AgNPs to bacteria, which causes damage to the latter [31].

5. Conclusions

We conclude from this study that silver nanoparticles can be made using a simple chemical reduction of silver salts, and that the granular sizes of the resulting silver nanoparticles are influenced by the reaction time and amount of reactants, with granular sizes increasing as the reaction time and amount of reactants increase. The results of electrospun nanofibers showed that the diameter of the fibers increases with increasing concentration and the stability of the other influencing factors, and that regular nanofibers can be obtained by selecting an appropriate concentration that is proportional to the other factors such as the applied voltage, the pumping ratio, and the needle diameter. The results of bacteria inhibition showed that a fabric made from polyvinyl chloride fibers and silver nanoparticles can inhibit both gram-positive and gram-negative bacteria, and that the efficiency of the inhibition rises as the concentration of silver nanoparticles in the tissue increases. We also note that its ability to suppress gram-negative bacteria is greater than that of gram-positive bacteria, but when the percentage of silver nanoparticles is increased to 0.7 percent, the inactivation effectiveness for both types of bacteria reaches 99.99 percent.

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